LABORATORY DATA AND DATA PACKAGE REVIEW FORM

(Technical and Administrative Review)

Project number: RP1581	Analyst: [redacted]	Reviewer: [redacted]
Project name: Freedom Industries		
Technique: GC/MS	PAC: [redacted]	Date submitted for review: 6/23/14 + 7/25/14

Administrative and Technical Review

	Data package includes:		Verification:
✓	Objective of work	✓	Proofread for content
✓	List of samples analyzed	✓	Check spelling and punctuation in text
✓	Method reference(s)	✓	Check for data transcription errors
✓	Description of sample prep/subsampling	✓	Check spelling of compounds/analytes
✓	Instrument identification	✓	Project number, analyst's initials and date on each NEIC generated record
✓	Software (include version number)	✓	Instrument Logbook entry by analyst
✓	QC requirements/data quality summary	✓	Reference to and description of tables
✓	Date of analysis		
✓	Summary of results		

Administrative and technical findings summary and actions requested, if any. Minor corrections, for example things noted by sticky note, need not be listed:*

Make correction noted in data package

Reviewer's signature: [redacted] **Date:** 8-5-14

Response to Action(s) Requested*

Made Corrections as discussed

Analyst's signature: [redacted] **Date:** 8-5-14

Final Statement of Reviewer

I have reviewed this data package and it meets the documentation requirements of the NEIC Quality System. The data support the results being reported.

Reviewer's signature: [redacted] **Date:** 8-5-14

I have presented a summary of QC data from these analyses to a Branch QA representative.
Data were provided by: email _____ hardcopy _____ LIMS X

Analyst's signature: [redacted] / 9-15-14 **Date:** 8-26-14 Revised LIMS to Report Date

*Attach additional sheets as [redacted]

Analysis Summary (Use n/a if item is not applicable to your analysis)

1	**Project Code**	RP1581
2	**Project Name**	Freedom Industries
3	**Project Location**	Charleston, West Virginia
4	**Analyst**	[redacted]
5	**Date of Summary**	July 23, 2014
6	**Time Frame of Analysis**	May thru June, 2014
7	**Objective of Analysis**	Analyze submitted tank samples for the presence of fatty acids
8	**Number of Samples/Aliquots for analysis**	One sample from each of two storage tanks (#393 and #203)
9	**Physical Phase(s) [how many of each phase]**	The sample from storage tank #393 was a single, semi-viscous liquid phase (L-1) that was opaque and dark brown in color. The sample from storage tank #203 consisted of a semi-viscous liquid phase (L-1) on top of a solid phase (S-2). Both phases were opaque and dark brown in color.
10	**Analysis Method /Procedure**	The current versions of these methodologies were used during these analyses: • *Organic Compound Analysis*, NEICPROC/00-049 • *EPA Method 8270 (used as guidance)*
11	**Sample Preparation Method/Procedure**	Replicate aliquots from each sample phase were placed in individual 20-mL clear glass vials. Six milliliters of an extraction solution (30% THF in IPA) was added to each vial, which was then hand shaken, vortex mixed for 10-15 seconds, sonicated for 5 minutes, then centrifuged for 5 minutes at 1500 rpm.
12	**Analysis Matrix/Solvent**	For each extraction solution, a 10 uL aliquot was combined with 990 uL of CH_2Cl_2 and the resulting solution was injected into the GC/MS system for analysis.
13	**Instrument Identification**	Agilent 7890A Gas Chromatograph – NEIC #5325 Agilent 5975C MSD- NEIC #5326 Column: HP-5MS UI, 20 m x 0.18 mm, 0.18 u (SN: USB540913H)
14	**Software Identification**	Chemstation E.02.00.493
15	**Instrument Method**	[redacted]_RP1581_Fatty Acids.M
16	**Additional Analysis Information**	Notebook pages: *Standard Preps*: 61466-61468, 61478-61479, 61481, 60290-60292 *Sample Analysis*: 61487-61495 *Method Development*: 61476-61477, 61480 *Qualitative & Preliminary Results*: 61483-61486
17	**Data Processing Method**	For each run sequence, see the accompanying Excel sheets, results entered into LIMS
18	**Under Scope of Accreditation (yes/No)?**	Yes

19	**Blank(s) evaluation (Yes/No)?**	*Yes-* No responses from solvent or extraction blanks were observed at the retention times of the target analytes.
20	**Initial Tune**	Prior to analyzing standards and samples, the instrument was auto-tuned in order to meet the recommended DFTPP criteria. Afterwards, a 50 ppm DFTPP solution in methylene chloride was analyzed and the fragmentation results were compared to the criteria listed in the current version of EPA method 8270. DFTPP solutions were analyzed prior to each run set. The fragmentation results from all of the DFTPP solution injections met the EPA 8270 criteria.
21	**Internal Standard Recovery**	No internal standards were used
22	**Surrogate Recovery**	No surrogate compounds were used
23	**Initial Calibration Verification**	Triplicate analysis of a second source (SS) standard solution produced acceptable analyte concentration values that validated the calibration curves used to quantify the target analytes. The mean % target values ranged from 82.0% to 113%.
24	**Continuing Calibration Verification (CCV)**	For each of the four run sequences, a CCV standard solution was prepared and dispensed into multiple sample vials. Thus, all CCV solution injections during a sample set run, came from un-punctured vials instead of multiple injections coming from the same vial. The observed analyte concentrations from all of these injections ranged from ranged from 86.8% to 119% indicating that the GC/MS system was working as expected.
25	**Replicate % Relative Standard Deviation**	Tank 393, L-1: rsd range- 0.87% to 20% Tank 203, L-1: rsd range- 1.1% to 11% Tank 203, S-2: rsd range- 0.77% to 10%
26	**Spike % Recovery**	Mean=102%, rsd=19%, n=30, range: 53.4% to 126%
27	**Control Samples % Recovery (or Deviation)**	n/a
28	**QC results outside of acceptance Criteria:**	None
29	**Approach for estimated Measurement Uncertainty**	Consulted with NEIC Statistician Dr. Brad Venner about estimating uncertainty for this data set. During these discussions, a few observations were made: 1. *CCV Data*: For three of the four run sets, the % target values from the CCV injections increased significantly during the course of these analyses. This trend, however, was not observed during the second source run set in which only standard solutions were analyzed. This suggests that the sample matrix may have been responsible for the increasing drift during these sample runs. The mean % target values

		observed during these three sets ranged from 16% to 19%. Trying to account for this run drift into an uncertainty evaluation would be rather difficult. 2. *Sample Run (6/20/14)*: The rsd values from the triplicate sample aliquots extractions were comparable to and in some case, better than the rsd values from the CCV solution injections. 3. *Matrix Spike Sample Runs (6/24 and 6/30):* An evaluation of the two data sets indicated no statistical difference of the analyte recovery data between the two MS samples. Thus, all 30 values were used to evaluate analyte recovery, which produced a mean value of 102%. Looking at the individual values, 28 fell within a recovery range of $\pm$ 30%. The two values outside of this range, the tetradecanoic and hexadecanoic acid recoveries from the Tank 393 MS sample were probably due to some matrix effects from the sample.
30	**Estimate of Measurement Uncertainty**	Based on the observations above and discussions with Dr. Venner, it was decided that a value of $\pm$ 30% would be an acceptable uncertainty estimate for the analyte content values reported in Table 1.
31	**Comment(s)**	Due to the high profile of this case, the analyte content results in Table 1 were reviewed and immediately sent afterwards to Special Agent Allison Landsman (see July 3, 2014 Memorandum). Review of the QC data was still pending at time the time this memo was sent. <u>8/5/14</u>: Review of the data package was completed the reviewer suggested that the MCHM and di-PPH results be based on area summations of the isomers rather than averaging individual isomer results, since this was the methodology used for the other MCHM and di-PPH analyses being done for this project. See the "Isomer Areas" worksheet for the recalculation of the MCHM and di-PPH content results. The attached *Narrative* discusses the method development work that was done prior to analyzing the tank samples, and the results presented in this section were not reported.

Table 1: Analyte Content Results by GC/MS

Compound Class	Analyte (# of carbon atoms in fatty chain)	Tank 393, L-1‡		Tank 203, L-1‡		Tank 203, S-2‡	
		Analyte Content*	Reporting Limit (R.L.)†	Analyte Content*	Reporting Limit (R.L.)†	Analyte Content*	Reporting Limit (R.L.)†
Saturated Fatty Acid	Octanoic Acid (C8)	1.88%	1.2%	1.17%	1.0%	1.30%	1.2%
	Decanoic Acid (C10)	1.12%	1.1%	<R.L.	1.0%	<R.L.	1.2%
	Dodecanoic Acid (C12)	1.99%	1.1%	1.29%	1.0%	1.44%	1.2%
	Tetradecanoic Acid (C14)	1.80%	1.2%	1.54%	1.0%	1.81%	1.2%
	Hexadecanoic Acid (C16)	2.46%	1.2%	2.72%	1.0%	5.34%	1.2%
	Octadecanoic Acid (C18)	<R.L.	1.1%	<R.L.	1.0%	<R.L.	1.2%
	Eicosanoic Acid (C20)	<R.L.	5.7%	<R.L.	5.0%	<R.L.	5.8%
Unsaturated Fatty Acid	Linoleic Acid (C18)	5.90%	2.8%	9.70%	2.5%	9.83%	2.9%
	Oleic Acid (C18)	5.80%	1.1%	6.51%	1.0%	6.21%	1.2%
MCHM Product	4-methylcyclohexanemethanol (MCHM)-Total**	0.203%	0.11%	0.339%	0.10%	0.307%	0.12%
	1-phenoxy-2-propanol (PPH)	<R.L.	0.11%	<R.L.	0.10%	<R.L.	0.12%
	1,4-cyclohexanedimethanol (1,4-CHDM)- Total**	<R.L.	0.11%	<R.L.	0.10%	<R.L.	0.12%
	Dimethylcyclohexane-1,4-dicarboxylate (DM-1,4-DC)	<R.L.	0.11%	<R.L.	0.10%	<R.L.	0.12%
	Dipropylene glycol phenyl ether (di-PPH)-Total**	0.201%	0.11%	0.521%	0.10%	0.490%	0.12%
Saturated Fatty Acid Methyl Ester	Octanoic acid methyl ester (C8)	0.110%	0.055%	0.0499%	0.048%	<R.L.	0.055%
	Decanoic acid methyl ester (C10)	<R.L.	0.092%	<R.L.	0.080%	<R.L.	0.093%
	Dodecanoic acid methyl ester (C12)	<R.L.	0.18%	<R.L.	0.16%	<R.L.	0.19%
	Tridecanoic Acid methyl ester (C13)	<R.L.	0.092%	<R.L.	0.080%	<R.L.	0.093%
	Tetradecanoic acid methyl ester (C14)	0.199%	0.092%	0.0980%	0.080%	0.0979%	0.093%
	Pentadecanoic Acid methyl ester (C15)	0.0548%	0.055%	<R.L.	0.048%	<R.L.	0.055%
	Hexadecanoic acid methyl ester (C16)	4.15%	0.373%	2.69%	0.33%	2.39%	0.38%
	Heptadecanoic Acid methyl ester (C17)	0.0975%	0.092%	<R.L.	0.080%	<R.L.	0.093%
	Octadecanoic acid methyl ester (C18)	1.54%	0.19%	0.914%	0.16%	0.834%	0.19%
	Eicosanoic Acid methyl ester (C20)	0.119%	0.055%	0.0919%	0.048%	0.0961%	0.055%
	Docosanoic Acid methyl ester (C22)	0.119%	0.055%	0.106%	0.048%	0.116%	0.055%
Unsaturated Fatty Acid Methyl Ester	Myristoleic Acid methyl ester (C14)	0.0650%	0.055%	<R.L.	0.048%	<R.L.	0.055%
	Palmitoleic Acid methyl ester (C16)	0.511%	0.18%	0.236%	0.16%	0.242%	0.19%
	Linoleic Acid methyl ester (C18)	9.76%	0.37%	9.31%	0.33%	8.42%	0.38%
	Linolenic Acid methyl ester (C18) Oleic Acid methyl ester (C18)	12.8%	0.75%	7.44%	0.65%	6.77%	0.76%
	Elaidic Acid methyl ester (C18)	0.874%	0.075%	0.405%	0.065%	0.343%	0.076%
	cis-11-Eicosenoic Acid methyl ester (C20)	0.179%	0.11%	0.125%	0.095%	0.129%	0.11%
	Erucic Acid methyl ester (C22)	<R.L.	0.055%	<R.L.	0.048%	<R.L.	0.055%

*-Analyte Contents are averaged values from replicate sample preparations. Because of the associated uncertainty, the three significant figures reported for these results do not reflect the accuracy of these values.

†-Reporting Limits were based on the analyte concentration in the lowest calibration standard and the sample preparation method

‡-Designation of "L" or "S" indicate a liquid or solid phase. The number indicates the phase order observed in the sample starting from the top (1) and moving downward.

**- "Total" designation indicates the analyte is comprised of two or more chemical isomers.

Narrative

Sample Analysis by Liquid Chromatography/Mass Spectrometry (LC/MS)

Initially, the analysis of the tanks samples was going to done using LC/MS because of the "soft" ionization technique preserves the parent molecule of a target analyte by adding or removing a hydrogen atom $[M+H]^+$ or $[M-H]^-$, or creating adducts with other molecular species (such as NH_4) to form an ionic species. Unlike other detectors, analytes detection by a mass spectrometer is based on the formation of charged masses.

Ion Trap Mass Spectrometer (4/22 to 5/2, 5/23/ to 5/30)

An ion trap mass spectrometer was initially used to evaluate analyte responses from individual and two fatty acid standard mixes. An atmospheric pressure chemical ionization (APCI) source was used because the formation of charged masses occurs within this source prior to introduction into the ion trap. Because of this, water and an organic solvent can be used as mobile phase components in the LC system without having to use any buffers or modifiers. For this work, methanol and water were used as mobile phase components. Individual solutions were infused into the mass spectrometer to evaluate and optimize analyte responses. Fatty acid mixes were injected into the LC system to evaluate and optimize the chromatographic separation of the target analytes. Analyte responses were observed in negative ion mode by the removal of a hydrogen atom to form the $[M-H]^-$ ion. The accuracy of the values assigned to these charged masses was accurate to 1 atomic mass unit (amu). This initial work evaluated a number of mobile phase gradients, three separation columns, chromatographic parameters including flow rate, column oven temp, etc. Having established some preliminary instrument parameters method development was then shifted to the LC/Quadrupole-Time-of-Flight (Q-ToF) mass spectrometer.

When the Q-ToF system went down because issues with the reference mass solution, the use of the ion trap system was re-evaluated. During this time, work was done using an electrospray ion source (ESI) along with the APCI source. (A number of journal articles discussed the use of an ESI source to analyze fatty acids.) Unlike the APCI source, using an ESI source requires that the charged masses need to be produced in solution prior to introduction into the ion trap, and so a number of mobile phase modifiers including 0.1% formic acid, 0.1% NH_4OAc and 0.1mM NH_4F were evaluated. Both sources were evaluated in positive and negative ion mode to see which source and mode produced the best analyte responses. As seem in previous work, analyte responses were only seen using the APCI source, in negative ion mode.

As mentioned earlier, the mass accuracy of an ion trap is good to 1 amu. Therefore, to confirm the presence of a target compound, the charged analyte needs to be fragmented and the resulting product ions and their abundances are compared to an analyte standard solution analyzed under the same instrument conditions. Unfortunately, fragmentation experiments using stearic acid did not generate any detectable product ions, suggesting the using this LC/ion trap system would not be feasible in analyzing the tank samples.

Quadrupole-Time-of-Flight (Q-ToF) Mass Spectrometer (5/8 to 5/20)

Using some instrument parameters from the initial work on the ion trap, method development work was shifted to Q-ToF mass spectrometer. Like the ion trap, analyte detection is based on charged masses. However, mass value observed for a target compound is accurate to one ten-thousandths of an amu, as opposed to the unit mass accuracy for the ion trap. The drawback to having this kind of mass accuracy, and hence analyte specificity, is that the detector is very sensitive to any temperature changes within the Q-ToF, and these slight changes can produce an inaccurate mass value for a target compound. Because of this, reference ions of known masses, are constantly infused into the Q-ToF in order compensate for any changes in the detector, and adjusts the mass values observed for a target analyte.

Initially, the two fatty acid mixes used for the ion trap work were injected into the LC/Q-ToF system. In addition, individual analyte solutions were prepared from two fatty acids kits; one acid kit contained neat material from saturated fatty acids ranging from C6 to C24 and the second kit contained neat materials of unsaturated fatty acids ranging from C16 to C24. A fatty acid methly ester (FAME) mix was also purchased. During the preparation of the C24 saturated fatty acid stock solution, a diluent comprised of a 30:70 THF:IPA solution was used to dissolve this neat material. Since this was the longest carbon chain acid, this THF:IPA solution was used as the diluent in the preparation of other acid stock solutions.

Two UPLC columns were evaluated along with a number of the LC parameters. Flow injections of a stearic acid solution were done to optimize some the MS parameters such as fragmentor and needle voltages, along with auto and targeted MS/MS experiments. Thomson vials that have a self filtration mechanism (injection solutions are passed through a filter disk on the bottom of a small polypropylene vial as it slides into a larger outer vial) were also evaluated. All of the saturated fatty acid stock solutions were further diluted and injected into the LC/Q-ToF system. Injections of a 1:1 IPA:MeOH solution was used to evaluate any analyte carry over between injections. A multimode source was initially set up to analyze these solutions in the APCI mode. This source was later set up as an ESI source to evaluate injections of the C24 fatty acid solution. Dilution experiments were run using tank extracts in order to determine the optimal extract volume to be used when analyzing the tank sample. During these analyses, however, the reference mass responses began to decrease to the point where their intensities were indistinguishable from background responses. Agilent field engineers were notified of this reference mass problem and arrived on site shortly after receiving a service call and were able to stabilize the reference mass responses. During these repairs, the engineers had difficulties in getting the instrument to meet their response criteria, and the troubleshooting as to why this was happening is still on-going.

Sample Analysis by Gas Chromatography/Mass Spectrometry (GC/MS)

Using Hydrogen as a Carrier Gas

With Q-ToF system being down and the ion trap unable to produce fragmentation data for these analytes it was decided to investigate the use of gas chromatography (GC) coupled with an MS detector to evaluate the tank samples. The first GC/MS system evaluated used hydrogen gas as a carrier gas. Individual solutions and analyte mixes (saturated and unsaturated fatty acids, and FAME) were injected into this GC/MS system as instrument parameters were evaluated. Using "Parameter Set #3" a 100 ppm glycerol solution, a 50 ppm saturated acid mix, a 50 ppm unsaturated acid mix and a 50 ppm FAME mix were injected and evaluated. Well defined peak responses were observed for the glycerol as well as the saturated acids and FAME analytes. Analyte responses from the unsaturated acid mix, however, were not as distinctive. For example, the five C18 unsaturated acid components eluted together with little chromatographic separation between these compounds. To evaluate their elution order, individual solutions of these compounds were injected and evaluated. Table 2 contains the observed retention times and mass spectral data (the three most abundant ions) for these analyte, along with the corresponding spectral data from the NIST library. The underlined values was the mass having the highest abundance for each mass set.

Table 2: C18 Unsaturated Fatty Acid Elution Order

		Mass Ions*	
C18 Unsaturated Acid Compound	Ret time(s) (min)	Observed	NIST Library
Linoleic Acid	**16.431**, ~16.6,~16.8, *17.312*	41,55,67	67,81,55
Linolenic Acid	**16.572**, ~16.8, *17.316*	79,41,55	41,79,67
Oleic Acid	**16.676**, 16.841, *17.335*	41,55,69	55,69,41
Petroselinic Acid	**16.808**, 16.935, *17.345*	41,55,69	41,55,69
Elaidic Acid *(trans isomer of Oleic acid)*	**16.907**, *17.340*	41,55,69	55,69,83
Stearic Acid (Saturated C18 acid)	*17.323*	41,55,73	43,73,60

*- Mass ions are from the peak responses with the bold face retention time value

As shown in Table 2, unsaturated acid response had some kind of peak splitting, as shown by the multiple of retention time values that are listed. The **bold face** retention time value was the tallest peak and assumed to be the target analyte. A comparison of the mass spectral pattern from these compounds obtained using the H_2 carrier gas were very similar to each other, but were noticeably different from the spectra in the NIST Library database, as shown in Table 2. Another observation, the last retention time listed for each compound was similar to the retention time of the saturated C18 fatty acid (stearic acid), and the mass spectra of these responses were similar to stearic acid as well. It is very possible that the H_2 carrier gas combined with the high temperatures and metal surfaces in the MS source, were adding H atoms across some of the C=C bonds and thus converting some of the C18 unsaturated acids to the saturated C18 acid. This hydrogenation reaction is well documented in many organic chemistry text books.

The analysis of diluted extract aliquots from Tank 393 and 203 indicated the presence of saturated and unsaturated fatty acid, and FAMEs and this information was shared with the case agent during a conference call on 6/11/14. Based on the investigations with the unsaturated fatty acid mix, were the saturated fatty acids and FAMEs observed in these diluted extracts, actually present in the tanks samples, or were they produced within the GC/MS system in which some unsaturated fatty acids and FAMEs were converted to saturated fatty acids and FAMEs.

The work done on this GC/MS system showed that the tank samples potentially contained saturated and unsaturated fatty acids along with FAMEs. However, quantifying these analytes using this GC/MS with the H_2 carrier gas could produce inaccurate results as to the composition of the tank samples because of the possible conversion of the unsaturated compounds to saturated compounds.

Using He as a Carrier Gas, Instrument Parameters and Qualitative Assessment

Method development work continued on a second GC/MS instrument that used helium (He) as its carrier gas. Initial solution injections did not produce acceptable peak responses for the target analytes and so a variety of maintenance functions (replacing injection port liner and gold seal, cleaning the MS source and changed out Merlin seal) were done to improve these analyte responses. Injections of a 50 ppm saturated fatty acid solution were then used to evaluate instrument parameters. However, sharp peak responses that were seen from this solution on the GC/MS with the H_2 carrier gas could not be reproduced on this system. The acid responses were noticeable lower to the point where the C22 and C24 acids did not produce any response. In an attempt to observe these two acids, the separation column was replace with another column having the same dimensions used in the H_2 system. However, no C22 or C24 acid responses were seen using this second column. Since these acids were not observed in the tank extracts that were analyzed on the first GC/MS system, method development continued.

Individual solutions of the saturated and unsaturated fatty acids, along with the FAMEs mix were injected to evaluate analyte retention times and the associated mass spectra. Unlike the previous GC/MS work, injections of the unsaturated C18 fatty acid solutions produced a single peak response, rather than a response having multiple peaks, and no stearic acid responses were observed in these solution injections. The analysis of the individual fatty acid solutions also contained a chromatographic response having a retention time of 7.46 minutes, which was similar to the retention time observed for dodecanoic acid methyl ester in the FAME mix. A library search its mass spectra identified this component as butylated hydroxytoluene, which is a stabilizing agent for THF, and THF was in the diluent used in preparing these stock solutions. Extracts of the tank samples were prepared and injected to determine the analytes present and during these analyses, components from the MCHM product formulation were identified. Therefore, a MCHM product solution standard was also injected in order to determine retention times and mass spectra of these analytes.

A quantitation method was initially set up based on the analytes observed in the tank extracts, and their responses (retention time, target and qual ions) observed from the various analyte solutions or mixes. A standard solution containing all of the analytes was prepared and injected and these quantitation parameters were evaluated. Many of these analytes eluted from the separation column such that an extracted ion chromatogram (EIC) based on the most abundance ion (i.e. base peak) could be used in quantifying these components, without any concerns of abundance contributions from nearby eluting compounds. There were, however, a few compounds where abundance contribution from nearby eluting compounds was of concern:

1. **Dipropylene glycol phenyl ether (Di-PPH) and dodecanoic acid**: there was sufficient separation between the first di-PPH isomer and dodecanoic acid that base peak (mass 59) could be used to quantify di-PPH (I). However, the dodecanoic acid response had a

lot of tailing such that the mass 59 abundance from this compound could significantly contribute to the mass 59 response for the other three di-PPH isomers. Further evaluation of the analyte spectra indicated that mass 94 could be used to quantify the three remaining di-PPH isomers with little or no abundance contribution for the dodecanoic acid.

2. **Linoleic/linolenic/oleic/elaidic acid methyl esters**: there was sufficient separation such that the base peaks for linoleic and elaidic acid methyl esters could be used to quantify these two compounds. As for linolenic and oleic acid methyl esters, these two compounds co-eluted with each other, and looking at their mass spectra, there would be a significant amount of abundance contributions to base peak responses between the two compounds. In fact, the only ions in which there would not any abundance contribution between the compounds were the molecular ions; mass 292 for the linolenic methyl ester and mass 296 for the oleic methyl ester. Based on this, these spectra were again evaluated to see if there were masses of similar abundances between the two analyte, which turned out to be mass 41.0 and mass 55.0. Therefore, the quantitation method, the 41 mass was entered a the target ion, with 55 being a qualifier ion for both analyte and both molecular ions were also included as qualifier ions, indicating that both compounds were presence. With that, the quantitation was setup such a viable response having a retention time of 14.18 min and having all four masses is due to linolenic methyl ester and oleic methyl ester, but calculating how much of each was present, could not be determined using the current instrument parameters.

3. **Octadecanoic acid methyl ester, linoleic and oleic acids**: the base peaks were suitable to quantifying these analytes. However, the separation between the linoleic and oleic acids was very poor and so manual integrations were needed in order to determine the best analyte responses for quantitation.

Based on these observations, changes to the quantitation method were made and used in the analysis of the tank samples.

The last parameter evaluated was the extract volume that would be diluted with CH_2Cl_2 and injected into the GC/MS system. Initially, 20 uL aliquots from the tank sample extracts were diluted to 1000 uL with CH_2Cl_2 and injected into the GC/MS. Looking at these chromatograms, a number of responses (known and unknown) were observed. One concern was that too much analyte material may have been injected onto the separation column. To evaluate this, a second set of diluted samples were prepared using 5 uL of the extracts. Although less material was injected onto the separation column, some of the compound observed in from the 20 uL aliquots solutions, were not seen, such as isomers II, III and IV for di-PPH. To obtain the best analyte responses without putting too much material on the separation column, an extract aliquot volume of 10 uL would be diluted to 1000 uL with CH_2Cl_2. With all of the instrument parameters in place, the analysis of the tank samples could begin.

OK

RP1581: Freedom Industries

Tank 393, L-1

[illegible] used the lowest RL

Sample: N405006-03: A — Amount (g): 0.1135; Extract Volume (mL): 6.00; Extract Aliquot (uL): 10.0; Final Inj Sol'n Volume (mL): 1.00

Analyte	[Analyte]$_{Lowest\ Cal\ Std}$ (ug/mL)	[Analyte]$_{inj\ sol'n}$ (ug/mL)	Amount in Sample (ug)[1]	Sample Content: Analyte[2]	Sample Content: Reporting Limit[3]	Group
Octanoic Acid	2.01	3.30	1980	1.74%	1.1%	
Decanoic Acid	2.00	1.86	<RL	<RL	1.1%	
Dodecanoic Acid	1.99	3.48	2088	1.84%	1.1%	
Tetradecanoic Acid	2.01	3.30	1980	1.74%	1.1%	
Hexadecanoic Acid	2.01	4.48	2688	2.37%	1.1%	17.0%
Linoleic Acid	4.96	8.74	5244	4.62%	2.6%	
Oleic Acid	1.98	8.80	5280	4.65%	1.0%	
Octadecanoic Acid	1.98	N.D.	<RL	<RL	1.0%	
Eicosanoic Acid	9.99	N.D.	<RL	<RL	5.3%	
MCHM (I)	*0.200*	*0.67*	*402*			
MCHM (II)	*0.200*	*0.20*	*120*			
Average MCHM *	0.200		261	0.230%	0.11%	
PPH	0.200	N.D.	<RL	------	0.11%	
1,4-CHDM (I)	*0.200*	*N.D.*	*<RL*			
1,4-CHDM (II)	*0.500*	*N.D.*	*<RL*			
Average 1,4-CHDM	0.200		<RL	------	0.11%	0.489%
DM-1,4-DC	0.200	N.D.	<RL	------	0.11%	
di-PPH (I)	*0.200*	*0.49*	*294*			
di-PPH (II)	*0.200*	*0.18*	*<RL*			
di-PPH (III)	*1.00*	*N.D.*	*<RL*			
di-PPH (IV)	*1.00*	*N.D.*	*<RL*			
Average di-PPH *	0.200		294	0.259%	0.11%	
Octanoic acid methyl ester	0.0950	0.20	120	0.106%	0.050%	
Decanoic acid methyl ester	0.160	0.14	<RL	<RL	0.085%	
Dodecanoic acid methyl ester	0.320	0.28	<RL	<RL	0.17%	
Tridecanoic Acid methyl ester	0.160	N.D.	<RL	<RL	0.085%	
Myristoleic Acid methyl ester (C14:1n9c)	0.0950	0.12	72	0.0634%	0.050%	
Tetradecanoic acid methyl ester	0.160	0.35	210	0.185%	0.085%	
Pentadecanoic Acid methyl ester	0.0950	N.D.	<RL	<RL	0.050%	
Palmitoleic Acid methyl ester (C16:1n9c)	0.320	0.91	546	0.481%	0.17%	
Hexadecanoic acid methyl ester	0.650	7.76	4656	4.10%	0.34%	
Heptadecanoic Acid methyl ester	0.160	0.17	102	0.0899%	0.085%	30.1%
Linoleic Acid methyl ester (C18:2n6c)	0.650	18.11	10866	9.57%	0.34%	
Linolenic Acid methyl ester (C18:3n3) Oleic Acid methyl ester (C18:1n9c) 296	1.30	23.99	14394	12.7%	0.69%	
Elaidic Acid methyl ester (C18:1n9t)	0.130	1.61	966	0.851%	0.069%	
Octadecanoic acid methyl ester	0.325	2.90	1740	1.53%	0.17%	
cis-11-Eicosenoic Acid methyl ester (C20:1)	0.190	0.31	186	0.164%	0.10%	
Eicosenoic Acid methyl ester	0.0950	0.22	132	0.116%	0.050%	
Erucic Acid methyl ester (C22:1n9)	0.0950	N.D.	<RL	<RL	0.050%	
Docosanoic Acid methyl ester	0.0950	0.22	132	0.116%	0.050%	
					Total	47.5%

Sample: N405006-03: B — Amount (g): 0.1148; Extract Volume (mL): 6.00; Extract Aliquot (uL): 10.0; Final Inj Sol'n Volume (mL): 1.00

Analyte	[Analyte]$_{inj\ sol'n}$ (ug/mL)	Amount in Sample (ug)[1]	Sample Content: Analyte[2]	Sample Content: Reporting Limit[3]	Group
Octanoic Acid	3.68	2208	1.92%	1.1%	
Decanoic Acid	2.06	1236	1.08%	1.0%	
Dodecanoic Acid	3.86	2316	2.02%	1.0%	
Tetradecanoic Acid	3.33	1998	1.74%	1.1%	
Hexadecanoic Acid	4.64	2784	2.43%	1.1%	21.7%
Linoleic Acid	11.84	7104	6.19%	2.6%	
Oleic Acid	12.05	7230	6.30%	1.0%	
Octadecanoic Acid	N.D.	<RL	<RL	1.0%	
Eicosanoic Acid	N.D.	<RL	<RL	5.2%	
MCHM (I)	*0.74*	*444*			
MCHM (II)	*0.24*	*144*			
Average MCHM		294	0.256%	0.10%	
PPH	0.09	<RL	------	0.10%	
1,4-CHDM (I)	*N.D.*	*<RL*			
1,4-CHDM (II)	*N.D.*	*<RL*			
Average 1,4-CHDM		<RL	------	0.10%	0.442%
DM-1,4-DC	N.D.	<RL	------	0.10%	
di-PPH (I)	*0.51*	*306*			
di-PPH (II)	*0.20*	*120*			
di-PPH (III)	*N.D.*	*<RL*			
di-PPH (IV)	*N.D.*	*<RL*			
Average di-PPH		213	0.186%	0.10%	
Octanoic acid methyl ester	0.21	126	0.110%	0.050%	
Decanoic acid methyl ester	0.15	<RL	<RL	0.084%	
Dodecanoic acid methyl ester	0.30	<RL	<RL	0.17%	
Tridecanoic Acid methyl ester	N.D.	<RL	<RL	0.084%	
Myristoleic Acid methyl ester (C14:1n9c)	0.12	72	0.0627%	0.050%	
Tetradecanoic acid methyl ester	0.39	234	0.204%	0.084%	
Pentadecanoic Acid methyl ester	0.10	60	0.0523%	0.050%	
Palmitoleic Acid methyl ester (C16:1n9c)	1.00	600	0.523%	0.17%	
Hexadecanoic acid methyl ester	8.01	4806	4.19%	0.34%	
Heptadecanoic Acid methyl ester	0.19	114	0.0993%	0.084%	30.8%
Linoleic Acid methyl ester (C18:2n6c)	18.94	11364	9.90%	0.34%	
Linolenic Acid methyl ester (C18:3n3) Oleic Acid methyl ester (C18:1n9c)	24.28	14568	12.7%	0.68%	
Elaidic Acid methyl ester (C18:1n9t)	1.86	1116	0.972%	0.068%	
Octadecanoic acid methyl ester	2.99	1794	1.56%	0.17%	
cis-11-Eicosenoic Acid methyl ester (C20:1)	0.36	216	0.188%	0.099%	
Eicosenoic Acid methyl ester	0.23	138	0.120%	0.050%	
Erucic Acid methyl ester (C22:1n9)	N.D.	<RL	<RL	0.050%	
Docosanoic Acid methyl ester	0.22	132	0.115%	0.050%	
				Total	52.9%

Sample: N405006-03: C — Amount (g): 0.1045; Extract Volume (mL): 6.00; Extract Aliquot (uL): 10.0; Final Inj Sol'n Volume (mL): 1.00

Analyte	[Analyte]$_{inj\ sol'n}$ (ug/mL)	Amount in Sample (ug)[1]	Sample Content: Analyte[2]	Sample Content: Reporting Limit[3]	Group
Octanoic Acid	3.46	2076	1.99%	1.2%	
Decanoic Acid	2.01	1206	1.15%	1.1%	
Dodecanoic Acid	3.68	2208	2.11%	1.1%	
Tetradecanoic Acid	3.31	1986	1.90%	1.2%	
Hexadecanoic Acid	4.53	2718	2.60%	1.2%	23.1%
Linoleic Acid	12.01	7206	6.90%	2.8%	
Oleic Acid	11.23	6738	6.45%	1.1%	
Octadecanoic Acid	N.D.	<RL	<RL	1.1%	
Eicosanoic Acid	N.D.	<RL	<RL	5.7%	
MCHM (I)	*0.68*	*408*			
MCHM (II)	*0.20*	*120*			
Average MCHM		264	0.253%	0.11%	
PPH	N.D.	<RL	------	0.11%	
1,4-CHDM (I)	*N.D.*	*<RL*			
1,4-CHDM (II)	*N.D.*	*<RL*			
Average 1,4-CHDM		<RL	------	0.11%	0.451%
DM-1,4-DC	N.D.	<RL	------	0.11%	
di-PPH (I)	*0.49*	*294*			
di-PPH (II)	*0.20*	*120*			
di-PPH (III)	*N.D.*	*<RL*			
di-PPH (IV)	*N.D.*	*<RL*			
Average di-PPH		207	0.198%	0.11%	
Octanoic acid methyl ester	0.20	120	0.115%	0.055%	
Decanoic acid methyl ester	0.14	<RL	<RL	0.092%	
Dodecanoic acid methyl ester	0.28	<RL	<RL	0.18%	
Tridecanoic Acid methyl ester	N.D.	<RL	<RL	0.092%	
Myristoleic Acid methyl ester (C14:1n9c)	0.12	72	0.0689%	0.055%	
Tetradecanoic acid methyl ester	0.36	216	0.207%	0.092%	
Pentadecanoic Acid methyl ester	0.10	60	0.0574%	0.055%	
Palmitoleic Acid methyl ester (C16:1n9c)	0.92	552	0.528%	0.18%	
Hexadecanoic acid methyl ester	7.25	4350	4.16%	0.37%	
Heptadecanoic Acid methyl ester	0.18	108	0.103%	0.092%	30.7%
Linoleic Acid methyl ester (C18:2n6c)	17.08	10248	9.81%	0.37%	
Linolenic Acid methyl ester (C18:3n3) Oleic Acid methyl ester (C18:1n9c)	22.43	13458	12.9%	0.75%	
Elaidic Acid methyl ester (C18:1n9t)	1.39	834	0.798%	0.075%	
Octadecanoic acid methyl ester	2.68	1608	1.54%	0.19%	
cis-11-Eicosenoic Acid methyl ester (C20:1)	0.32	192	0.184%	0.11%	
Eicosenoic Acid methyl ester	0.21	126	0.121%	0.055%	
Erucic Acid methyl ester (C22:1n9)	N.D.	<RL	<RL	0.055%	
Docosanoic Acid methyl ester	0.22	132	0.126%	0.055%	
				Total	54.2%

Overall Sample Content (%)

Analyte	Analyte: mean	Analyte: s	Analyte: rsd	Analyte: Reporting Limit[4]	Group
Octanoic Acid	1.88%	0.13%	6.7%	1.2%	
Decanoic Acid	**1.12%**	------	------	1.1%	
Dodecanoic Acid	1.99%	0.14%	7.0%	1.1%	
Tetradecanoic Acid	1.80%	0.091%	5.1%	1.2%	
Hexadecanoic Acid	2.46%	0.12%	4.9%	1.2%	21.0%
Linoleic Acid	5.90%	1.2%	20%	2.8%	
Oleic Acid	5.80%	1.0%	17%	1.1%	
Octadecanoic Acid	<R.L.	------	------	1.1%	
Eicosanoic Acid	<R.L.	------	------	5.7%	
MCHM (I)					
MCHM (II)					
Average MCHM	0.246%	0.014%	5.8%	0.11%	
PPH	<R.L.	------	------	0.11%	
1,4-CHDM (I)					
1,4-CHDM (II)					
Average 1,4-CHDM	<R.L.	------	------	0.11%	0.46%
DM-1,4-DC	<R.L.	------	------	0.11%	
di-PPH (I)					
di-PPH (II)					
di-PPH (III)					
di-PPH (IV)					
Average di-PPH	0.214%	0.039%	18%	0.11%	
Octanoic acid methyl ester	0.110%	0.0046%	4.1%	0.055%	
Decanoic acid methyl ester	<R.L.	------	------	0.092%	
Dodecanoic acid methyl ester	<R.L.	------	------	0.18%	
Tridecanoic Acid methyl ester	<R.L.	------	------	0.092%	
Myristoleic Acid methyl ester (C14:1n9c)	0.0650%	0.0034%	5.2%	0.055%	
Tetradecanoic acid methyl ester	0.199%	0.012%	5.9%	0.092%	
Pentadecanoic Acid methyl ester	**0.0548%**	------	------	0.055%	
Palmitoleic Acid methyl ester (C16:1n9c)	0.511%	0.026%	5.0%	0.18%	
Hexadecanoic acid methyl ester	4.15%	0.043%	1.0%	0.37%	
Heptadecanoic Acid methyl ester	0.0975%	0.0069%	7.1%	0.092%	30.5%
Linoleic Acid methyl ester (C18:2n6c)	9.76%	0.17%	1.7%	0.37%	
Linolenic Acid methyl ester (C18:3n3) Oleic Acid methyl ester (C18:1n9c)	12.8%	0.11%	0.87%	0.75%	
Elaidic Acid methyl ester (C18:1n9t)	0.874%	0.089%	10%	0.075%	
Octadecanoic acid methyl ester	1.54%	0.016%	1.0%	0.19%	
cis-11-Eicosenoic Acid methyl ester (C20:1)	0.179%	0.0129%	7.2%	0.11%	
Eicosenoic Acid methyl ester	0.119%	0.0024%	2.0%	0.055%	
Erucic Acid methyl ester (C22:1n9)	<R.L.	------	------	0.055%	
Docosanoic Acid methyl ester	0.119%	0.0062%	5.2%	0.055%	
				Total	51.9%

* - Results not reported see "Isomer Areas" worksheet, [redacted] 8/5/14

1- Analyte amount in sample (ug)= $\frac{[Analyte]_{inj\ sol'n}\ (ug/mL) \times (1.00\ mL_{inj\ sol'n})}{[Extract\ Aliquot(uL) \times (1.00\ mL/1000\ uL)]} \times$ Extract Volume (mL)

2- Analyte % = $\frac{\text{Analyte amount (ug)} \times (1\ g/1{,}000{,}000\ ug)}{\text{Sample Amount (g)}} \times 100\%$

3- Reporting Limit [RL] (%) = $\frac{[Analyte]_{Lowest\ Cal\ Sol'n}\ (ug/mL) \times (1.00\ mL_{inj\ sol'n})}{[Extract\ Aliquot(uL) \times (1.00\ mL/1000\ uL)]} \times \text{Extract Volume (mL)} \times \frac{1\ g}{1{,}000{,}000\ ug} \times \frac{100\%}{\text{Sample Amount (g)}}$

4- Overall Analyte Reporting Limit (%) = The largest RL value from the analyte replicates.

Note:

1. Results for the linolenic and oleic acid methyl esters of were combined into the single report value because:
   A. The compounds could not be separated chromatographically with the instrument parameters used.
   B. Although the molecular ions for both compounds were observed, no other qualitative masses unique to each analyte were observed.
2. The overall sample content values for decanoic acid and pentadecanoic acid methyl ester were averages from two individual results.

7-31-14 [redacted]

Reviewed [illegible]

NOTE: For MCHM, 1,4-CHDM and di-PPH, the observed analyte amount in the sample was the averaged value from the isomer responses that were greater than their low cal std response.

[redacted] 7/2/14

File : Fatty Acid Sample Analysis.xlsx, *Worksheet* : Sample Analysis

RP1581: Freedom Industries

		Tank 203, L-1																			
		Sample: N405006-05: A		Amount (g): 0.1263; Extract Volume (mL): 6.00; Extract Aliquot (uL): 10.0; Final Inj Sol'n Volume (mL): 1.00			Sample: N405006-05: B		Amount (g): 0.1401; Extract Volume (mL): 6.00; Extract Aliquot (uL): 10.0; Final Inj Sol'n Volume (mL): 1.00			Sample: N405006-05: C		Amount (g): 0.1195; Extract Volume (mL): 6.00; Extract Aliquot (uL): 10.0; Final Inj Sol'n Volume (mL): 1.00			Overall Sample Content (%)				
				Sample Content					Sample Content					Sample Content			Analyte				
Analyte	$[Analyte]_{Lowest\ Cal\ Std}$ (ug/mL)	$[Analyte]_{inj\ sol'n}$ (ug/mL)	Amount in Sample (ug)[1]	Analyte[2]	Reporting Limit[3]	Group	$[Analyte]_{inj\ sol'n}$ (ug/mL)	Amount in Sample (ug)[1]	Analyte[2]	Reporting Limit[3]	Group	$[Analyte]_{inj\ sol'n}$ (ug/mL)	Amount in Sample (ug)[1]	Analyte[2]	Reporting Limit[3]	Group	mean	s	rsd	Reporting Limit[4]	Group
Octanoic Acid	2.01	2.43	1458	1.15%	0.95%		2.63	1578	1.13%	0.86%		2.43	1458	1.22%	1.0%		1.17%	0.048%	4.1%	1.0%	
Decanoic Acid	2.00	1.57	<RL	<RL	0.95%		1.66	<RL	<RL	0.86%		1.58	<RL	<RL	1.0%		<R.L.	------	------	1.0%	
Dodecanoic Acid	1.99	2.69	1614	1.28%	0.95%		2.85	1710	1.22%	0.85%		2.72	1632	1.37%	1.0%		1.29%	0.073%	5.7%	1.0%	
Tetradecanoic Acid	2.01	3.29	1974	1.56%	0.95%		3.32	1992	1.42%	0.86%		3.28	1968	1.65%	1.0%		1.54%	0.11%	7.4%	1.0%	
Hexadecanoic Acid	2.01	5.56	3336	2.64%	0.95%	20.9%	6.20	3720	2.66%	0.86%	23.8%	5.70	3420	2.86%	1.0%	24.1%	2.72%	0.12%	4.5%	1.0%	22.9%
Linoleic Acid	4.96	17.87	10722	8.49%	2.4%		24.29	14574	10.4%	2.1%		20.32	12192	10.2%	2.5%		9.70%	1.05%	11%	2.5%	
Oleic Acid	1.98	12.09	7254	5.74%	0.94%		16.39	9834	7.02%	0.85%		13.45	8070	6.75%	0.99%		6.51%	0.67%	10%	1.0%	
Octadecanoic Acid	1.98	N.D.	<RL	<RL	0.94%		N.D.	<RL	<RL	0.85%		N.D.	<RL	<RL	0.99%		<R.L.	------	------	1.0%	
Eicosanoic Acid	9.99	N.D.	<RL	<RL	4.7%		N.D.	<RL	<RL	4.3%		N.D.	<RL	<RL	5.0%		<R.L.	------	------	5.0%	
MCHM (I)	*0.200*	*1.31*	*786*				*1.46*	*876*				*1.29*	*774*								
MCHM (II)	*0.200*	*0.41*	*246*				*0.49*	*294*				*0.35*	*210*								
Average MCHM *	0.200		516	0.409%	0.095%			585	0.418%	0.086%			492	0.412%	0.10%		0.413%	0.0046%	1.1%	0.10%	
PPH	0.200	0.17	<RL	------	0.095%		0.21	126	0.0899%	0.086%		0.18	<RL	------	0.10%		<R.L.	------	------	0.10%	
1,4-CHDM (I)	*0.200*	*N.D.*	*<RL*				*N.D.*	*<RL*				*N.D.*	*<RL*								
1,4-CHDM (II)	*0.500*	*N.D.*	*<RL*				*N.D.*	*<RL*				*N.D.*	*<RL*								
Average 1,4-CHDM	0.200		<RL	------	0.095%	1.1%		<RL	------	0.086%	1.2%		<RL	------	0.10%	1.3%	<R.L.	------	------	0.10%	1.2%
DM-1,4-DC	0.200	N.D.	<RL	------	0.095%		N.D.	<RL	------	0.086%		N.D.	<RL	------	0.10%		<R.L.	------	------	0.10%	
di-PPH (I)	*0.200*	*1.08*	*648*				*1.27*	*762*				*1.13*	*678*								
di-PPH (II)	*0.200*	*0.79*	*474*				*0.94*	*564*				*0.89*	*534*								
di-PPH (III)	*1.00*	*1.74*	*1044*				*2.06*	*1236*				*2.00*	*1200*								
di-PPH (IV)	*1.00*	*2.32*	*1392*				*2.65*	*1590*				*2.87*	*1722*								
Average di-PPH *	0.200		889.5	0.704%	0.095%			1038	0.741%	0.086%			1033.5	0.865%	0.10%		0.770%	0.084%	11%	0.10%	
Octanoic acid methyl ester	0.0950	0.11	66	0.0523%	0.045%		0.11	66	0.0471%	0.041%		0.10	60	0.0502%	0.048%		0.0499%	0.0026%	5.2%	0.048%	
Decanoic acid methyl ester	0.160	N.D.	<RL	<RL	0.076%		N.D.	<RL	<RL	0.069%		N.D.	<RL	<RL	0.080%		<R.L.	------	------	0.080%	
Dodecanoic acid methyl ester	0.320	0.03	<RL	<RL	0.15%		0.04	<RL	<RL	0.14%		0.02	<RL	<RL	0.16%		<R.L.	------	------	0.16%	
Tridecanoic Acid methyl ester	0.160	N.D.	<RL	<RL	0.076%		N.D.	<RL	<RL	0.069%		N.D.	<RL	<RL	0.080%		<R.L.	------	------	0.080%	
Myristoleic Acid methyl ester (C14:1n9c)	0.0950	N.D.	<RL	<RL	0.045%		N.D.	<RL	<RL	0.041%		N.D.	<RL	<RL	0.048%		<R.L.	------	------	0.048%	
Tetradecanoic acid methyl ester	0.160	0.20	120	0.0950%	0.076%		0.23	138	0.0985%	0.069%		0.20	120	0.100%	0.080%		0.0980%	0.0027%	2.8%	0.080%	
Pentadecanoic Acid methyl ester	0.0950	N.D.	<RL	<RL	0.045%		N.D.	<RL	<RL	0.041%		N.D.	<RL	<RL	0.048%		<R.L.	------	------	0.048%	
Palmitoleic Acid methyl ester (C16:1n9c)	0.320	0.51	306	0.242%	0.15%		0.56	336	0.240%	0.14%		0.45	270	0.226%	0.16%		0.236%	0.0088%	3.7%	0.16%	
Hexadecanoic acid methyl ester	0.650	5.56	3336	2.64%	0.31%		6.41	3846	2.75%	0.28%		5.35	3210	2.69%	0.33%		2.69%	0.052%	1.9%	0.33%	
Heptadecanoic Acid methyl ester	0.160	0.16	96	0.0760%	0.076%	21.1%	0.16	96	0.0685%	0.069%	21.8%	0.16	96	0.0803%	0.080%	21.7%	<R.L.	------	------	0.080%	21.5%
Linoleic Acid methyl ester (C18:2n6c)	0.650	19.32	11592	9.18%	0.31%		21.95	13170	9.40%	0.28%		18.65	11190	9.36%	0.33%		9.31%	0.12%	1.3%	0.33%	
Linolenic Acid methyl ester (C18:3n3) Oleic Acid methyl ester (C18:1n9c) 296	1.30	15.15	9090	7.20%	0.62%		17.77	10662	7.61%	0.56%		14.94	8964	7.50%	0.65%		7.44%	0.21%	2.88%	0.65%	
Elaidic Acid methyl ester (C18:1n9t)	0.130	0.84	504	0.399%	0.062%		0.91	546	0.390%	0.056%		0.85	510	0.427%	0.065%		0.405%	0.019%	4.8%	0.065%	
Octadecanoic acid methyl ester	0.325	1.88	1128	0.893%	0.15%		2.17	1302	0.929%	0.14%		1.83	1098	0.919%	0.16%		0.914%	0.019%	2.0%	0.16%	
cis-11-Eicosenoic Acid methyl ester (C20:1)	0.190	0.28	168	0.133%	0.090%		0.27	162	0.116%	0.081%		0.25	150	0.126%	0.095%		0.125%	0.0087%	7.0%	0.095%	
Eicosenoic Acid methyl ester	0.0950	0.19	114	0.0903%	0.045%		0.21	126	0.0899%	0.041%		0.19	114	0.0954%	0.048%		0.0919%	0.0031%	3.3%	0.048%	
Erucic Acid methyl ester (C22:1n9)	0.0950	N.D.	<RL	<RL	0.045%		N.D.	<RL	<RL	0.041%		N.D.	<RL	<RL	0.048%		<R.L.	------	------	0.048%	
Docosanoic Acid methyl ester	0.0950	0.22	132	0.105%	0.045%		0.24	144	0.103%	0.041%		0.22	132	0.110%	0.048%		0.106%	0.0040%	3.8%	0.048%	
					Total	43.1%				Total	46.9%				Total	47.0%				Total	45.6%

* - Results not reported, see "Isomer Areas" worksheet [redacted] 8/5/14

[1]- Analyte amount in sample (ug)= $\frac{[Analyte]_{inj\ sol'n}\ (ug/mL) \times (1.00\ mL_{inj\ sol'n})}{[Extract\ Aliquot(uL) \times (1.00\ mL/1000\ uL)]} \times$ Extract Volume (mL)

[2]- Analyte % = $\frac{Analyte\ amount\ (ug) \times (1\ g/1{,}000{,}000\ ug)}{Sample\ Amount\ (g)} \times 100\%$

[3]- Reporting Limit [RL] (%) = $\frac{[Analyte]_{Lowest\ Cal\ Std'n}\ (ug/mL) \times (1.00\ mL_{inj\ sol'n})}{[Extract\ Aliquot(uL) \times (1.00\ mL/1000\ uL)]} \times$ Extract Volume (mL) $\times \frac{1\ g}{1{,}000{,}000\ ug} \times \frac{100\%}{Sample\ Amount\ (g)}$

[4]- Overall Analyte Reporting Limit (%) = The largest RL value from the analyte replicates.

NOTE: For MCHM, 1,4-CHDM and di-PPH, the observed analyte amount in the sample was the averaged value from the isomer responses that were greater than their low cal std response.

Note:

1. Results for the linolenic and oleic acid methyl esters of were combined into the single report value because:
   A. The compounds could not be separated chromatographically with the instrument parameters used.
   B. Although the molecular ions for both compounds were observed, no other qualitative masses unique to each analyte were observed.

7-31-14 [redacted]

Reviewed 7/31/14 [redacted]

[redacted] 7/2/14

File : Fatty Acid Sample Analysis.xlsx, *Worksheet* : Sample Analysis

81: Freedom Industries

OK

Tank 203, S-2												
06: B — Amount (g): 0.1028; Extract Volume (mL): 6.00; Extract Aliquot (uL): 10.0; Final Inj Sol'n Volume (mL): 1.00			Sample: N405006-06: C — Amount (g): 0.1095; Extract Volume (mL): 6.00; Extract Aliquot (uL): 10.0; Final Inj Sol'n Volume (mL): 1.00					Overall Sample Content (%)				
Sample Content			[Analyte]inj sol'n	Amount in	Sample Content			Analyte				
Analyte[2]	Reporting Limit[3]	Group	(ug/mL)	Sample (ug)[1]	Analyte[2]	Reporting Limit[3]	Group	mean	s	rsd	Reporting Limit[4]	Group
1.34%	1.2%		2.35	1410	1.29%	1.1%		1.30%	0.044%	3.4%	1.2%	
<RL	1.2%		1.53	<RL	<RL	1.1%		<R.L.	------	------	1.2%	
1.51%	1.2%		2.62	1572	1.44%	1.1%		1.44%	0.072%	5.0%	1.2%	
1.91%	1.2%		3.26	1956	1.79%	1.1%		1.81%	0.085%	4.7%	1.2%	
4.93%	1.2%	26.4%	9.35	5610	5.12%	1.1%	26.1%	5.34%	0.542%	10%	1.2%	25.9%
10.1%	2.9%		18.47	11082	10.1%	2.7%		9.83%	0.490%	5.0%	2.9%	
6.58%	1.2%		11.57	6942	6.34%	1.1%		6.21%	0.441%	7.1%	1.2%	
<RL	1.2%		N.D.	<RL	<RL	1.1%		<R.L.	------	------	1.2%	
<RL	5.8%		N.D.	<RL	<RL	5.5%		<R.L.	------	------	5.8%	
			1.10	*660*								
			0.35	*210*								
0.371%	0.12%			435	0.397%	0.11%		0.377%	0.018%	4.9%	0.12%	
------	0.12%		0.17	<RL	------	0.11%		<R.L.	------	------	0.12%	
			N.D.	*<RL*								
			N.D.	*<RL*								
------	0.12%	1.03%		<RL	------	0.11%	1.15%	<R.L.	------	------	0.12%	1.05%
------	0.12%		N.D.	<RL	------	0.11%		<R.L.	------	------	0.12%	
			0.96	*576*								
			0.72	*432*								
			1.68	*1008*								
			2.13	*1278*								
0.661%	0.12%			823.5	0.752%	0.11%		0.676%	0.070%	10%	0.12%	
<RL	0.055%		0.09	<RL	<RL	0.052%		<R.L.	------	------	0.055%	
<RL	0.093%		N.D.	<RL	<RL	0.088%		<R.L.	------	------	0.093%	
<RL	0.19%		0.01	<RL	<RL	0.18%		<R.L.	------	------	0.19%	
<RL	0.093%		N.D.	<RL	<RL	0.088%		<R.L.	------	------	0.093%	
<RL	0.055%		N.D.	<RL	<RL	0.052%		<R.L.	------	------	0.055%	
0.0992%	0.093%		0.18	108	0.0986%	0.088%		0.0979%	0.0018%	1.9%	0.093%	
<RL	0.055%		N.D.	<RL	<RL	0.052%		<R.L.	------	------	0.055%	
0.257%	0.19%		0.46	276	0.252%	0.18%		0.242%	0.021%	8.7%	0.19%	
2.38%	0.38%		4.36	2616	2.39%	0.36%		2.39%	0.018%	0.77%	0.38%	
------	0.093%	19.2%	0.16	96	0.0877%	0.088%	19.7%	<R.L.	------	------	0.093%	19.4%
8.29%	0.38%		15.59	9354	8.54%	0.36%		8.42%	0.127%	1.5%	0.38%	
6.71%	0.76%		12.44	7464	6.82%	0.71%		6.77%	0.056%	0.83%	0.76%	
0.339%	0.076%		0.59	354	0.323%	0.071%		0.343%	0.022%	6.5%	0.076%	
0.811%	0.19%		1.54	924	0.844%	0.18%		0.834%	0.020%	2.4%	0.19%	
0.134%	0.11%		0.23	138	0.126%	0.10%		0.129%	0.0043%	3.3%	0.11%	
0.0992%	0.055%		0.18	108	0.0986%	0.052%		0.0961%	0.0049%	5.1%	0.055%	
<RL	0.055%		N.D.	<RL	<RL	0.052%		<R.L.	------	------	0.055%	
0.123%	0.055%		0.21	126	0.115%	0.052%		0.116%	0.0055%	4.7%	0.055%	
	Total	46.6%				Total	46.9%				Total	46.4%

Note:

1. Results for the linolenic and oleic acid methyl esters of were combined into the single report value because:
   A. The compounds could not be separated chromatographically with the instrument parameters used.
   B. Although the molecular ions for both compounds were observed, no other qualitative masses unique to each analyte were observed.

_ x 100%

0 ug   Sample Amount (g)

7-31-14

[redacted]

Reviewed

[redacted]

from the isomer responses that were greater than their low cal std response.

RP1581: Freedom Industries

Fatty Acids Results

Compound Class	Analyte (# of carbon atoms in fatty chain)	Tank 393, L-1‡		Tank 203, L-1‡		Tank 203, S-2‡	
		Analyte Content*	Reporting Limit (R.L.)†	Analyte Content*	Reporting Limit (R.L.)†	Analyte Content*	Reporting Limit (R.L.)†
Saturated Fatty Acid	Octanoic Acid (C8)	1.88%	1.2%	1.17%	1.0%	1.30%	1.2%
	Decanoic Acid (C10)	1.12%	1.1%	<R.L.	1.0%	<R.L.	1.2%
	Dodecanoic Acid (C12)	1.99%	1.1%	1.29%	1.0%	1.44%	1.2%
	Tetradecanoic Acid (C14)	1.80%	1.2%	1.54%	1.0%	1.81%	1.2%
	Hexadecanoic Acid (C16)	2.46%	1.2%	2.72%	1.0%	5.34%	1.2%
	Octadecanoic Acid (C18)	<R.L.	1.1%	<R.L.	1.0%	<R.L.	1.2%
	Eicosanoic Acid (C20)	<R.L.	5.7%	<R.L.	5.0%	<R.L.	5.8%
Unsaturated Fatty Acid	Linoleic Acid (C18)	5.90%	2.8%	9.70%	2.5%	9.83%	2.9%
	Oleic Acid (C18)	5.80%	1.1%	6.51%	1.0%	6.21%	1.2%
MCHM Product	MCHM	0.246%	0.11%	0.413%	0.10%	0.377%	0.12%
	PPH	<R.L.	0.11%	<R.L.	0.10%	<R.L.	0.12%
	1,4-CHDM	<R.L.	0.11%	<R.L.	0.10%	<R.L.	0.12%
	DM-1,4-DC	<R.L.	0.11%	<R.L.	0.10%	<R.L.	0.12%
	di-PPH	0.214%	0.11%	0.770%	0.10%	0.676%	0.12%
Saturated Fatty Acid Methyl Ester	Octanoic acid methyl ester (C8)	0.110%	0.055%	0.0499%	0.048%	<R.L.	0.055%
	Decanoic acid methyl ester (C10)	<R.L.	0.092%	<R.L.	0.080%	<R.L.	0.093%
	Dodecanoic acid methyl ester (C12)	<R.L.	0.18%	<R.L.	0.16%	<R.L.	0.19%
	Tridecanoic Acid methyl ester (C13)	<R.L.	0.092%	<R.L.	0.080%	<R.L.	0.093%
	Tetradecanoic acid methyl ester (C14)	0.199%	0.092%	0.0980%	0.080%	0.0979%	0.093%
	Pentadecanoic Acid methyl ester (C15)	0.0548%	0.055%	<R.L.	0.048%	<R.L.	0.055%
	Hexadecanoic acid methyl ester (C16)	4.15%	0.373%	2.69%	0.33%	2.39%	0.38%
	Heptadecanoic Acid methyl ester (C17)	0.0975%	0.092%	<R.L.	0.080%	<R.L.	0.093%
	Octadecanoic acid methyl ester (C18)	1.54%	0.19%	0.914%	0.16%	0.834%	0.19%
	Eicosanoic Acid methyl ester (C20)	0.119%	0.055%	0.0919%	0.048%	0.0961%	0.055%
	Docosanoic Acid methyl ester (C22)	0.119%	0.055%	0.106%	0.048%	0.116%	0.055%
Unsaturated Fatty Acid Methyl Ester	Myristoleic Acid methyl ester (C14)	0.0650%	0.055%	<R.L.	0.048%	<R.L.	0.055%
	Palmitoleic Acid methyl ester (C16)	0.511%	0.18%	0.236%	0.16%	0.242%	0.19%
	Linoleic Acid methyl ester (C18)	9.76%	0.37%	9.31%	0.33%	8.42%	0.38%
	Linolenic Acid methyl ester (C18) Oleic Acid methyl ester (C18)	12.8%	0.75%	7.44%	0.65%	6.77%	0.76%
	Elaidic Acid methyl ester (C18)	0.874%	0.075%	0.405%	0.065%	0.343%	0.076%
	cis-11-Eicosenoic Acid methyl ester (C20)	0.179%	0.11%	0.125%	0.095%	0.129%	0.11%
	Erucic Acid methyl ester (C22)	<R.L.	0.055%	<R.L.	0.048%	<R.L.	0.055%

* Analyte Content are averaged values from replicate sample preparations

†-Reporting Limits were based on the analyte concentration in the lowest calibration standard and the sample preparation method

‡-Designation of "L" or "S" indicate a liquid or solid phase. The number indicates the phase order observed in the sample starting from the top (1) and moving downward.

Note: Results for the linolenic and oleic acid methyl esters of were combined into the single report value because:

A. The compounds could not be separated chromatographically with the instrument parameters used.
B. Although the molecular ions for both compounds were observed, no other qualitative masses unique to each analyte were observed.

File: Fatty Acid Sample Analysis-7-16.xlsx, Worksheet: Results_7-2-14

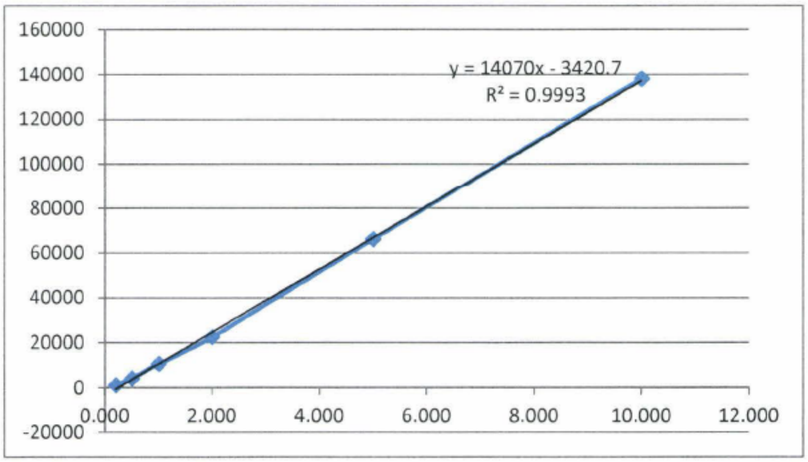
[redacted] RP1581, 8/19/14

RP1581: Freedom Industries

Di-PPH Calculations based on the combined isomer area responses

Calibration Standards

Standard	[di-PPH] (ug/ml)	di-PPH Isomer Area I	II	III	IV	Total
Std 1	0.200	464	714	0	0	1178
Std 2	0.500	1824	2288	0	0	4112
Std 3	1.00	4824	4835	560	308	10527
Std 4	2.00	11307	9848	831	562	22548
Std 5	5.00	36007	26410	2299	1459	66175
Std 6	10.0	77931	51800	5611	2695	138037



Calculated concentations: [Analyte] (ug/mL) = (Area Response - y-intercept)/slope

$[\text{di-PPH}]_{\sum \text{isomer areas}}$ (ug/mL) = [di-PPH total isomer area - (-3420.7)]/14070

Sample	di-PPH Isomer Areas I	II	III	IV	Total	$[\,]_{\sum \text{isomer areas}}$ (ug/mL)	Sample Amount (g)	% Sample $\sum$ isomer_{Area} [1]	% Sample Ave Isomer $_{Amt}$ [2]	Relative % Diff
N405006-03:	1064	584	0	0	1648	0.360	0.1135	0.190%	0.259%	-31%
N405006-03:	1221	706	0	0	1927	0.380	0.1148	0.199%	0.186%	6.6%
N405006-03:	1097	701	0	0	1798	0.371	0.1045	0.213%	0.198%	7.3%
							Ave	0.201%	0.214%	-6.6%
N405006-05:	5788	3808	748	674	11018	1.026	0.1263	0.488%	0.704%	-36%
N405006-05:	7284	4601	934	763	13582	1.208	0.1401	0.518%	0.741%	-36%
N405006-05:	6185	4299	899	821	12204	1.110	0.1195	0.558%	0.865%	-43%
							Ave	0.521%	0.770%	-39%
N405006-06:	4658	3167	555	486	8866	0.873	0.1127	0.465%	0.615%	-28%
N405006-06:	4614	2915	585	464	8578	0.853	0.1028	0.498%	0.661%	-28%
N405006-06:	4865	3412	718	625	9620	0.927	0.1095	0.508%	0.752%	-39%
							Ave	0.490%	0.676%	-32%

[1]- % Sample based on $\sum$ isomer_{Area} = [illegible]$]_{\sum \text{isomerArea}}$ (ug/mL) X (1.00 $mL_{inj\ sol'n}$/0.0100 $mL_{ext\ sol'n}$) x (6.00 $mL_{ext\ sol'n}$ /1,000,000 ug)}/sample amount (g) x 100%

[2]- % Sample based on Average Isomer amount, see "Sample Analysis" Worksheet.

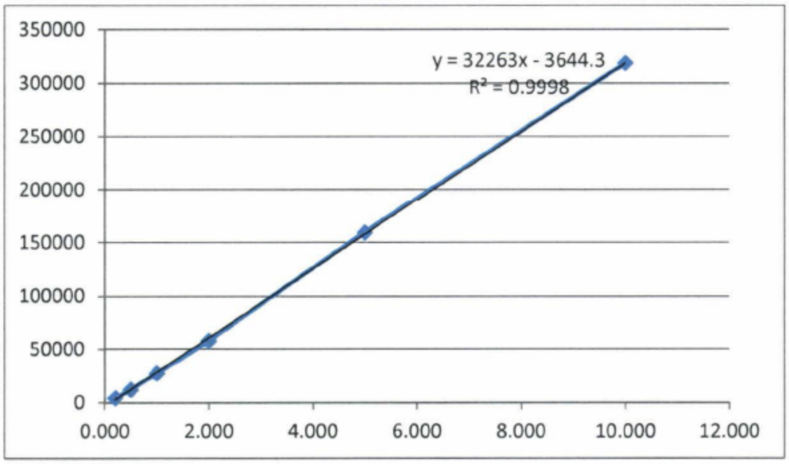
Since there is significant difference (RPD) between the average values of the $\sum$ isomer_{Area} and Ave Isomer_{Amt} the $\sum$ isomer_{Area} will be reported since this calculation method was also being used to determine MCHM contents in other project samples.

RP1581: Freedom Industries

MCHM Calculations based on the combined isomer area responses

Calibration Standards

Standard	[MCHM] (ug/ml)	MCHM Isomer Area I	II	Total
Std 1	0.200	1584	3114	4698
Std 2	0.500	4124	8418	12542
Std 3	1.00	9024	18888	27912
Std 4	2.00	18815	39341	58156
Std 5	5.00	50528	108963	159491
Std 6	10.0	106376	212269	318645



Calculated concentations: [Analyte] (ug/mL) = (Area Response - y-intercept)/slope

$[MCHM]_{\sum isomer\ areas}$ (ug/mL) = [MCHM total isomer area - (-3644.3)]/32263

Sample	MCHM Isomer Areas I	II	Total	$[]_{\sum isomer\ areas}$ (ug/mL)	Sample Amount (g)	% MCHM Sample $\sum$ isomer$_{Area}$ [1]	Ave Isomer $_{Amt}$ [2]	Relative % Diff
N405006-03:A	5562	2323	7885	0.357	0.1135	0.189%	0.230%	-20%
N405006-03:B	6314	3134	9448	0.406	0.1148	0.212%	0.256%	-19%
N405006-03:C	5662	2428	8090	0.364	0.1045	0.209%	0.253%	-19%
					Ave	0.203%	0.246%	-19%
N405006-05:A	12413	6794	19207	0.708	0.1263	0.336%	0.409%	-19%
N405006-05:B	13989	8662	22651	0.815	0.1401	0.349%	0.418%	-18%
N405006-05:C	12170	5514	17684	0.661	0.1195	0.332%	0.412%	-22%
					Ave	0.339%	0.413%	-20%
N405006-06:A	9765	4358	14123	0.551	0.1127	0.293%	0.362%	-21%
N405006-06:B	8971	4084	13055	0.518	0.1028	0.302%	0.371%	-20%
N405006-06:C	10076	5485	15561	0.595	0.1095	0.326%	0.397%	-20%
					Ave	0.307%	0.377%	-20%

[1]- % Sample based on $\sum$ isomer$_{Area}$ = {[]$_{\sum isomerArea}$ (ug/mL) X (1.00 mL$_{inj\ sol'n}$/0.0100 mL$_{ext\ sol'n}$) x (6.00 mL$_{ext\ sol'n}$ /1,000,000 ug)}/sample amount (g) x 100%

[2]- % Sample based on Average Isomer amount, see "Sample Analysis" Worksheet.

Since there is significant difference (RPD) between the average values of the $\sum$ isomer$_{Area}$ and Ave Isomer$_{Amt}$ the $\sum$ isomer$_{Area}$ will be reported since this calculation method was also being used to determine MCHM contents in other project samples.

RP1581: Freedom Industries

Fatty Acids Results

Compound Class	Analyte (# of carbon atoms in fatty chain)	Tank 393, L-1‡ Analyte Content*	Tank 393, L-1‡ Reporting Limit (R.L.)†	Tank 203, L-1‡ Analyte Content*	Tank 203, L-1‡ Reporting Limit (R.L.)†	Tank 203, S-2‡ Analyte Content*	Tank 203, S-2‡ Reporting Limit (R.L.)†
Saturated Fatty Acid	Octanoic Acid (C8)	1.88%	1.2%	1.17%	1.0%	1.30%	1.2%
	Decanoic Acid (C10)	1.12%	1.1%	<R.L.	1.0%	<R.L.	1.2%
	Dodecanoic Acid (C12)	1.99%	1.1%	1.29%	1.0%	1.44%	1.2%
	Tetradecanoic Acid (C14)	1.80%	1.2%	1.54%	1.0%	1.81%	1.2%
	Hexadecanoic Acid (C16)	2.46%	1.2%	2.72%	1.0%	5.34%	1.2%
	Octadecanoic Acid (C18)	<R.L.	1.1%	<R.L.	1.0%	<R.L.	1.2%
	Eicosanoic Acid (C20)	<R.L.	5.7%	<R.L.	5.0%	<R.L.	5.8%
Unsaturated Fatty Acid	Linoleic Acid (C18)	5.90%	2.8%	9.70%	2.5%	9.83%	2.9%
	Oleic Acid (C18)	5.80%	1.1%	6.51%	1.0%	6.21%	1.2%
MCHM Product	MCHM	0.203%	0.11%	0.339%	0.10%	0.307%	0.12%
	PPH	<R.L.	0.11%	<R.L.	0.10%	<R.L.	0.12%
	1,4-CHDM	<R.L.	0.11%	<R.L.	0.10%	<R.L.	0.12%
	DM-1,4-DC	<R.L.	0.11%	<R.L.	0.10%	<R.L.	0.12%
	di-PPH	0.201%	0.11%	0.521%	0.10%	0.490%	0.12%
Saturated Fatty Acid Methyl Ester	Octanoic acid methyl ester (C8)	0.110%	0.055%	0.0499%	0.048%	<R.L.	0.055%
	Decanoic acid methyl ester (C10)	<R.L.	0.092%	<R.L.	0.080%	<R.L.	0.093%
	Dodecanoic acid methyl ester (C12)	<R.L.	0.18%	<R.L.	0.16%	<R.L.	0.19%
	Tridecanoic Acid methyl ester (C13)	<R.L.	0.092%	<R.L.	0.080%	<R.L.	0.093%
	Tetradecanoic acid methyl ester (C14)	0.199%	0.092%	0.0980%	0.080%	0.0979%	0.093%
	Pentadecanoic Acid methyl ester (C15)	0.0548%	0.055%	<R.L.	0.048%	<R.L.	0.055%
	Hexadecanoic acid methyl ester (C16)	4.15%	0.373%	2.69%	0.33%	2.39%	0.38%
	Heptadecanoic Acid methyl ester (C17)	0.0975%	0.092%	<R.L.	0.080%	<R.L.	0.093%
	Octadecanoic acid methyl ester (C18)	1.54%	0.19%	0.914%	0.16%	0.834%	0.19%
	Eicosanoic Acid methyl ester (C20)	0.119%	0.055%	0.0919%	0.048%	0.0961%	0.055%
	Docosanoic Acid methyl ester (C22)	0.119%	0.055%	0.106%	0.048%	0.116%	0.055%
Unsaturated Fatty Acid Methyl Ester	Myristoleic Acid methyl ester (C14)	0.0650%	0.055%	<R.L.	0.048%	<R.L.	0.055%
	Palmitoleic Acid methyl ester (C16)	0.511%	0.18%	0.236%	0.16%	0.242%	0.19%
	Linoleic Acid methyl ester (C18)	9.76%	0.37%	9.31%	0.33%	8.42%	0.38%
	Linolenic Acid methyl ester (C18) Oleic Acid methyl ester (C18)	12.8%	0.75%	7.44%	0.65%	6.77%	0.76%
	Elaidic Acid methyl ester (C18)	0.874%	0.075%	0.405%	0.065%	0.343%	0.076%
	cis-11-Eicosenoic Acid methyl ester (C20)	0.179%	0.11%	0.125%	0.095%	0.129%	0.11%
	Erucic Acid methyl ester (C22)	<R.L.	0.055%	<R.L.	0.048%	<R.L.	0.055%

* Analyte Content are averaged values from replicate sample preparations

†-Reporting Limits were based on the analyte concentration in the lowest calibration standard and the sample preparation method

‡-Designation of "L" or "S" indicate a liquid or solid phase. The number indicates the phase order observed in the sample starting from the top (1) and moving downward.

Note: Results for the linolenic and oleic acid methyl esters of were combined into the single report value because:

A. The compounds could not be separated chromatographically with the instrument parameters used.

B. Although the molecular ions for both compounds were observed, no other qualitative masses unique to each analyte were observed.

Results in red are based on the summation of the isomer areas (see "Isomer Areas" worksheet)

File: Fatty Acid Sample Analysis-7-16.xlsx, Worksheet: Final Results_8-5-14

[redacted] RPP/RBi, 8/19/14

1

Date: 8/6/14

To: RP1581 Fatty Acid File

From: [redacted] , [redacted]

Re: Second Source Standard Solution

The second source analysis done during the fatty acid analyses used a second dilution of the FAME mix and a second set of weighings from the same neat materials used in preparing the calibration standards. Since analyte material from another source was not used, this solution is really a second weighing standard rather than a second source standard.

RP1581: Freedom Industries

Preparation and Run Date: 6/18/14 **(see p. 61487-61488)**

Solution Mix	Aliquot (uL) Std 1	Std 2	Std 3	Std 4	Std 5	Std 6
100 ppm FA	20	50	75	100	200	400
100 ppm MCHM	2.0	5.0	10	20	50	100
1000 ppm FAME	5.0	10	25	50	75	150
CH_2Cl_2	973	935	890	830	675	350
Total	1000	1000	1000	1000	1000	1000

Solution Mix	Analyte	Weight %	$[Analyte]_{mix}$ (ug/mL)	$[Analyte]_{Cal\ Std}$ (ug/mL) Std 1	Std 2	Std 3	Std 4	Std 5	Std 6
100 ppm Fatty Acids (FA)	Octanoic Acid		100.4	2.01	5.02	7.53	10.04	20.08	40.16
	Decanoic Acid		99.90	2.00	5.00	7.49	9.99	19.98	39.96
	Dodecanoic Acid		99.74	1.99	4.99	7.48	9.97	19.95	39.90
	Tetradecanoic Acid		100.6	2.01	5.03	7.55	10.06	20.12	40.24
	Hexadecanoic Acid		100.5	2.01	5.03	7.54	10.05	20.10	40.20
	Linoleic Acid		99.18	1.98	4.96	7.44	9.92	19.84	39.67
	Oleic Acid		98.82	1.98*	4.94	7.41	9.88	19.76	39.53
	Octadecanoic Acid		99.19	1.98	4.96	7.44	9.92	19.84	39.68
	Eicosanoic Acid		99.92	2.00	5.00	7.49	9.99	19.98	39.97
100 ppm MCHM	MCHM		100.0	0.200	0.500	1.000	2.000	5.000	10.00
	PPH		100.0	0.200	0.500	1.000	2.000	5.000	10.00
	1,4-CHDM		100.0	0.200	0.500	1.000	2.000	5.000	10.00
	DMCH-1,4-DC		100.0	0.200	0.500	1.000	2.000	5.000	10.00
	di-PPH		100.0	0.200	0.500	1.000	2.000	5.000	10.00
1000 ppm Fatty Acid Methyl Esters (FAME)**	Octanoic Acid Methyl Ester	1.9%	19.00	0.0950	0.190	0.475	0.950	1.425	2.850
	Decanoic Acid Methyl Ester	3.2%	32.00	0.160	0.320	0.800	1.600	2.400	4.800
	Dodecanoic Acid Methyl Ester	6.4%	64.00	0.320	0.640	1.600	3.200	4.800	9.600
	Tridecanoic Acid Methyl Ester	3.2%	32.00	0.160	0.320	0.800	1.600	2.400	4.800
	Myristoleic Acid Methyl Ester	1.9%	19.00	0.0950	0.190	0.475	0.950	1.425	2.850
	Tetradecanoic Acid Methyl Ester	3.2%	32.00	0.160	0.320	0.800	1.600	2.400	4.800
	Pentadecanoic Acid Methyl Ester	1.9%	19.00	0.0950	0.190	0.475	0.950	1.425	2.850
	Palmitoleic Acid, Methyl Ester	6.4%	64.00	0.320	0.640	1.600	3.200	4.800	9.600
	Hexadecanoic Acid Methyl Ester	13.0%	130.0	0.650	1.300	3.250	6.500	9.750	19.50
	Heptadecanoic Acid Methyl Ester	3.2%	32.00	0.160	0.320	0.800	1.600	2.400	4.800
	Linoleic Acid Methyl Ester	13.0%	130.0	0.650	1.300	3.250	6.500	9.750	19.50
	Linolenic Acid Methyl Ester	6.4%	*Combined concentrations since these analytes could not be separated chromatographically with the instrument parameters used or by unique masses.*						
	Oleic Acid Methyl Ester	19.6%							
	Linolenic/Oleic Acid Methyl Esters	*26.0%*	*260.0*	*1.300*	*2.600*	*6.500*	*13.00*	*19.50*	*39.00*
	Elaidic Acid Methyl Ester	2.6%	26.00	0.130	0.260	0.650	1.300	1.950	3.900
	Octadecanoic Acid Methyl Ester	6.5%	65.00	0.325	0.650	1.625	3.250	4.875	9.750
	cis-11-Eicosenoic Acid Methyl Ester	1.9%	19.00	0.0950	0.190	0.475	0.950	1.425	2.850
	Eicosanoic Acid Methyl Ester	1.9%	19.00	0.0950	0.190	0.475	0.950	1.425	2.850
	Erucic Acid Methyl Ester	1.9%	19.00	0.0950	0.190	0.475	0.950	1.425	2.850
	Docosanoic Acid Methyl Ester	1.9%	19.00	0.0950	0.190	0.475	0.950	1.425	2.850

**- For the fatty acid methyl esters, the % values were taken from the C of A for the Supleco mix, Lot LC00126

$$[Analyte]_{Cal\ Std}\ (ug/mL) = \frac{\text{Solution Mix Aliquot (uL)} \times [Analyte]_{mix}\ (ug/ml)}{\text{Volume Calibration Standard (uL)}}$$

For Cal Std 1:

$$[\text{Octanoic Acid}]\ (ug/mL) = \frac{100.4\ ug/mL \times 20\ uL}{1000\ uL} = 2.01\ ug/mL$$

$$[\text{Octanoic Acid Methyl Ester}]\ (ug/mL) = \frac{1000\ ug/mL_{FAME\ Mix} \times (1.9\%/100\%)_{Wt.\%\ Octanoic\ ME} \times 5.00\ uL}{1000\ uL} = 0.0950\ ug/mL$$

(b) [redacted] 7) 8/6/14

Notes: *Oleic Acid-* Although the oleic acid concentration in cal std 1 was 1.98 ug/mL, a value of 2.00 ug/mL was inadvertantly entered into the calibration data section of the instrument method. This error was discovered during the data review. However, a comparison of the observed oleic acid concentration in a CCV std based on this cal curve , and a second curve that used a 1.98 value, exhibited a relative percent difference value of less than 0.1% between the two curves. Therefore, the results based on the 2.00 ug/mL concentration value were still valid and will be reported.

Linoleic Acid Methyl Ester- A weight % value of 13% was used in calculating the analyte concentrations in the calibration standards. During a second data review, the reviewer noted that the weight % value list on the Certificate of Analysis for this analyte was actually 12.8%. With this, a second calibration curve in which the concentrations of this methyl ester were based on 12.8% was generated, and using the linear regression parameters from this curve, a linoleic acid methyl ester concentration was calculated for and compared to the result based on the original calibration curve. Since the % difference of the second curve to the first curve is 2%, the results based on the original curve are considered valid and will be reported.

Eicosanoic Acid- During the calibration data set up for the instrument method, eicosanoic acid responses were only observed in cal stds 4,5 and 6. A three point linear regression analysis produced an r^2 value of 0.975 indicating that quantifying eiconsanoic acid with the instrument

Results comparisons:

1.) Between calibration curves using 1.98 ug/mL and 2.00 ug/mL values for oleic acid in cal std 1.

Calibration Curve: Quadratic Regression

Response = $ax^2 + bx + c$, where x= [Analyte] (ug/mL)

2.00 ug/mL:	a =	6.83E+01	
	b =	3.92E+03	r^2=0.994
	c =	-1.49E+04	
1.98 ug/mL:	a =	6.85E+01	
	b =	3.91E+03	r^2=0.994
	c =	-1.48E+04	

$$x = \frac{(-b + \{SQRT(b^2-[4a(c-Area)]\})}{2a}$$

The oleic acid area response of the first CCV Std from the 6/20/14 run: 23571

	2.00 ug/mL	1.98 ug/mL	RPD
[Oleic Acid] (ug/mL)	8.543	8.537	0.067%

2.) Between linoleic acid methyl ester calibration curves based on 13% and 12.8% weight values.

For N405006-03: A

Linoleic acid methyl ester area 269600

[Linoleic acid methyl ester] (ug/mL):	18.11	Original curve using 13% value
	17.79	Second curve using 12.8% value
% Difference	-1.8%	

[redacted] 8/6/14

RP1581: Freedom Industries

Continuing Calibration Verification (CCV) Evaluation

Mix	Analyte		Mix Preparation: [Analyte]stk (ug/mL)	Mix Preparation: Stk Sol'n Aliquot (uL)	Mix Preparation: [Analyte]Mix (ug/mL)	CCV Standard Preparation: Mix Aliquot (uL)	CCV Standard Preparation: [Analyte]known (ug/mL)	Sample Analysis, Run Date: 6/20/14 — CCV Std Inj #1: [Analyte]obs (ug/mL)	CCV Std Inj #1: % of Target	CCV Std Inj #2: [Analyte]obs (ug/mL)	CCV Std Inj #2: % of Target	CCV Std Inj #3: [Analyte]obs (ug/mL)	CCV Std Inj #3: % of Target	CCV Std Inj #4: [Analyte]obs (ug/mL)	CCV Std Inj #4: % of Target	% of Target$_{n=4}$: mean	% of Target$_{n=4}$: s	% of Target$_{n=4}$: rsd	Run Drift[1]
Fatty Acids (see p.61488)	Octanoic Acid		6477	15.5	100.4	100	10.0	9.20	91.6%	9.95	99.1%	10.14	101%	11.03	110%	100%	7.5%	7.5%	18%
	Decanoic Acid		3330	30.0	99.9		10.0	8.83	88.4%	10.99	110%	11.12	111%	12.23	122%	108%	14%	13%	34%
	Dodecanoic Acid		2891	34.5	99.7		10.0	9.30	93.3%	9.85	98.8%	10.52	106%	11.05	111%	102%	7.7%	7.5%	18%
	Tetradecanoic Acid (a.k.a. Myristic)		3047	33.0	100.6		10.1	8.79	87.4%	9.60	95.4%	9.89	98.3%	10.98	109%	97.6%	9.0%	9.2%	22%
	Hexadecanoic Acid (a.k.a. Palmitic)		2645	38.0	100.5		10.1	7.64	76.0%	8.75	87.1%	9.05	90.0%	9.84	97.9%	87.8%	9.1%	10%	22%
	Linoleic Acid		6011	16.5	99.2		9.92	7.49	75.5%	8.92	89.9%	9.87	99.5%	10.01	101%	91.5%	12%	13%	25%
	Oleic Acid		5201	19.0	98.8		9.88	8.54	86.4%	9.62	97.4%	10.12	102%	10.28	104%	97.6%	7.9%	8.1%	18%
	Octadecanoic Acid (a.k.a. Stearic)		4133	24.0	99.2		9.92	9.27	93.4%	9.45	95.3%	9.39	94.7%	9.45	95.3%	94.7%	0.86%	0.90%	1.8%
	Eicosanoic Acid		3843	26.0	99.9		10.0	N.D.	------	N.D.	------	N.D.	------	N.D.	------	-----	-----	-----	-----
	CH_2Cl_2			763.5															
MCHM Product (from A. Hunter, see p.60290-60292)	*MCHM (I)*					20.0		*1.96*		*2.19*		*2.21*		*2.38*					
	MCHM (II)							*2.14*		*2.15*		*2.31*		*2.51*					
	Average MCHM		10000	10.0	100.0		2.00	2.05	103%	2.17	109%	2.26	113%	2.45	122%	112%	8.3%	7.5%	20%
	PPH		10000	10.0	100.0		2.00	2.12	106%	2.34	117%	2.34	117%	2.49	125%	116%	7.6%	6.6%	19%
	1,4-CHDM (I)							*2.19*		*2.51*		*2.76*		*2.92*					
	1,4-CHDM (II)							*1.99*		*2.28*		*2.36*		*2.49*					
	1,4-CHDM		10000	10.0	100.0		2.00	2.09	105%	2.40	120%	2.56	128%	2.71	135%	122%	13%	11%	31%
	DM-1,4-DC		10000	10.0	100.0		2.00	2.17	109%	2.42	121%	2.35	118%	2.54	127%	119%	7.7%	6.5%	19%
	di-PPH (I)							*1.86*		*2.08*		*2.25*		*2.46*					
	di-PPH (II)							*2.07*		*2.27*		*2.32*		*2.36*					
	di-PPH (III)							*2.04*		*2.30*		*2.36*		*2.14*					
	di-PPH (IV)							*2.22*		*2.51*		*2.48*		*2.32*					
	di-PPH		10000	10.0	100.0		2.00	2.05	102%	2.29	115%	2.35	118%	2.32	116%	113%	7.0%	6.2%	14%
	CH_2Cl_2			950															
FAMEs Mix (Supelco, Lot# LC00126) [FAME] (mg/mL): 10.0 (Prepared on 6/18/14)	Octanoic acid methyl ester	1.9%	190	100.0	19.0	50.0	0.95	0.98	103%	1.09	115%	1.06	112%	1.12	118%	112%	6.3%	5.7%	15%
	Decanoic acid methyl ester	3.2%	320		32.0		1.60	1.62	101%	1.84	115%	1.78	111%	1.89	118%	111%	7.3%	6.6%	17%
	Dodecanoic acid methyl ester	6.4%	640		64.0		3.20	3.53	110%	3.75	117%	3.81	119%	4.02	126%	118%	6.3%	5.3%	15%
	Tridecanoic Acid methyl ester	3.2%	320		32.0		1.60	1.71	107%	1.84	115%	1.83	114%	1.93	121%	114%	5.6%	4.9%	14%
	Myristoleic Acid methyl ester (C14:1n9c)	1.9%	190		19.0		0.95	0.97	102%	1.00	105%	1.03	108%	1.11	117%	108%	6.3%	5.9%	15%
	Tetradecanoic acid methyl ester	3.2%	320		32.0		1.60	1.72	108%	1.80	113%	1.84	115%	1.91	119%	114%	5.0%	4.4%	12%
	Pentadecanoic Acid methyl ester	1.9%	190		19.0		0.95	0.97	102%	1.02	107%	1.05	111%	1.10	116%	109%	5.7%	5.3%	14%
	Palmitoleic Acid methyl ester (C16:1n9c)	6.4%	640		64.0		3.20	3.31	103%	3.42	107%	3.44	108%	3.67	115%	108%	4.7%	4.4%	11%
	Hexadecanoic acid methyl ester	13.0%	1300		130		6.50	6.98	107%	7.23	111%	7.22	111%	7.62	117%	112%	4.1%	3.6%	9.8%
	Heptadecanoic Acid methyl ester	3.2%	320		32.0		1.60	1.60	100%	1.69	106%	1.69	106%	1.83	114%	106%	5.9%	5.6%	14%
	Linoleic Acid methyl ester (C18:2n6c)	13.0%	1300		130		6.50	6.54	101%	6.89	106%	7.02	108%	7.29	112%	107%	4.8%	4.5%	12%
	Linolenic Acid methyl ester (C18:3n3)- 6.4% Oleic Acid methyl ester (C18:1n9c)- 19.6%	26.0%	2600		260		13.00	14.12	109%	14.71	113%	14.74	113%	15.12	116%	113%	3.2%	2.8%	7.7%
	Elaidic Acid methyl ester (C18:1n9t)	2.6%	260		26.0		1.30	1.29	99.2%	1.36	105%	1.40	108%	1.46	112%	106%	5.5%	5.2%	13%
	Octadecanoic acid methyl ester	6.5%	650		65.0		3.25	3.34	103%	3.54	109%	3.52	108%	3.66	113%	108%	4.1%	3.8%	10%
	cis-11-Eicosenoic Acid methyl ester (C20:1)	1.9%	190		19.0		0.95	0.95	100%	0.98	103%	0.94	98.9%	1.01	106%	102%	3.3%	3.3%	6.3%
	Eicosenoic Acid methyl ester	1.9%	190		19.0		0.95	0.93	97.9%	0.94	98.9%	0.96	101%	1.01	106%	101%	3.7%	3.7%	8.4%
	Erucic Acid methyl ester (C22:1n9)	1.9%	190		19.0		0.95	0.86	90.5%	0.91	95.8%	0.91	95.8%	1.03	108%	97.6%	7.6%	7.8%	18%
	Docosanoic Acid methyl ester	1.9%	190		19.0		0.95	0.91	95.8%	0.92	96.8%	1.00	105%	1.01	106%	101%	5.5%	5.4%	11%
	CH_2Cl_2			900.0		830.0													

min=	87.8%	mean	16%
max=	122%	s	6.7%
		rsd	43%

1 - Run Drift = % Target Value$_{last\ CCV}$ - % Target Value$_{first\ CCV}$

2 - RDP = Relative Percent Difference = abs[%target$_{second\ CCV}$ - %target$_{first\ CCV}$]/Average % Target

8-1-14 [redacted]

[redacted] 6/30/14

RP1581: Freedom Industries

Continuing Calibration Verification (CCV) Evaluation

Mix	Analyte		Mix Preparation: $[Analyte]_{Stk}$ (ug/mL)	Mix Preparation: Stk Sol'n Aliquot (uL)	Mix Preparation: $[Analyte]_{Mix}$ (ug/mL)	CCV Standard Preparation: Mix Aliquot (uL)	CCV Standard Preparation: $[Analyte]_{known}$ (ug/mL)	Matrix Spike #1, Run Date: 6/24/14 — CCV Std Inj #1: $[Analyte]_{obs}$ (ug/mL)	CCV Std Inj #1: % of Target	CCV Std Inj #2: $[Analyte]_{obs}$ (ug/mL)	CCV Std Inj #2: % of Target	Average % Target	RPD[2]	Run Drift[1]	Second Source Evaluation, Run Date: 6/25/14 — CCV Std Inj #1: $[Analyte]_{obs}$ (ug/mL)	CCV Std Inj #1: % of Target	CCV Std Inj #2: $[Analyte]_{obs}$ (ug/mL)	CCV Std Inj #2: % of Target	CCV Std Inj #3: $[Analyte]_{obs}$ (ug/mL)	CCV Std Inj #3: % of Target	% of $Target_{n=3}$: mean	% of $Target_{n=3}$: s	% of $Target_{n=3}$: rsd	Run Drift[1]
Fatty Acids (see p.61488)	Octanoic Acid		6477	15.5	100.4		10.0	8.12	80.9%	10.62	106%	93.3%	27%	25%	9.29	92.5%	9.57	95.3%	9.68	96.4%	94.8%	2.0%	2.1%	3.9%
	Decanoic Acid		3330	30.0	99.9		10.0	8.01	80.2%	10.14	102%	90.8%	23%	21%	9.97	99.8%	9.63	96.4%	10.59	106%	101%	4.9%	4.8%	6.2%
	Dodecanoic Acid		2891	34.5	99.7		10.0	8.36	83.9%	10.82	109%	96.2%	26%	25%	9.84	98.7%	9.57	96.0%	9.87	99.0%	97.9%	1.7%	1.7%	0.3%
	Tetradecanoic Acid (a.ka. Myristic)		3047	33.0	100.6		10.1	7.87	78.2%	9.94	98.8%	88.5%	23%	21%	9.07	90.2%	9.07	90.2%	9.31	92.5%	91.0%	1.4%	1.5%	2.4%
	Hexadecanoic Acid (a.k.a. Palmitic)		2645	38.0	100.5	100	10.1	7.17	71.3%	9.03	89.9%	80.6%	23%	19%	7.94	79.0%	8.02	79.8%	8.38	83.4%	80.7%	2.3%	2.9%	4.4%
	Linoleic Acid		6011	16.5	99.2		9.92	7.28	73.4%	9.58	96.6%	85.0%	27%	23%	8.01	80.7%	7.79	78.5%	9.32	94.0%	84.4%	8.3%	9.9%	13%
	Oleic Acid		5201	19.0	98.8		9.88	7.52	76.1%	9.65	97.7%	86.9%	25%	22%	8.15	82.5%	8.50	86.0%	8.86	89.7%	86.1%	3.6%	4.2%	7.2%
	Octadecanoic Acid (a.k.a. Stearic)		4133	24.0	99.2		9.92	8.39	84.6%	8.39	84.6%	84.6%	0.0%	0.0%	7.76	78.2%	7.94	80.0%	9.16	92.3%	83.5%	7.7%	9.2%	14%
	Eicosanoic Acid		3843	26.0	99.9		10.0	11.70	------	N.D.	------	------	------	------	N.D.	------	N.D.	------	N.D.	------	-----	-----	-----	-----
	CH_2Cl_2			763.5																				
MCHM Product (from A. Hunter, see p.60290-60292)	*MCHM (I)*							1.76		2.28					1.95		1.97		2.01					
	MCHM (II)							1.85		2.21					2.08		2.12		2.07					
	Average MCHM		10000	10.0	100.0		2.00	1.81	90.3%	2.25	112%	101.3%	22%	22%	2.02	101%	2.05	102%	2.04	102%	102%	0.80%	0.79%	1.3%
	PPH		10000	10.0	100.0		2.00	1.81	90.5%	2.18	109%	99.8%	19%	19%	2.10	105%	2.04	102%	2.11	106%	104%	1.9%	1.8%	0.5%
	1,4-CHDM (I)							1.92		2.53					2.11		1.99		1.91					
	1,4-CHDM (II)							1.83		2.25					2.13		2.09		2.04					
	1,4-CHDM		10000	10.0	100.0		2.00	1.88	93.8%	2.39	120%	106.6%	24%	26%	2.12	106%	2.04	102%	1.98	98.8%	102%	3.6%	3.6%	-7.3%
	DM-1,4-DC		10000	10.0	100.0	20.0	2.00	1.92	96.0%	2.27	114%	104.8%	17%	18%	2.17	109%	2.13	107%	2.21	111%	109%	2.0%	1.8%	2.0%
	di-PPH (I)							1.60		2.28					1.84		1.88		1.94					
	di-PPH (II)							1.88		2.12					2.12		2.05		2.11					
	di-PPH (III)							2.21		2.32					2.37		2.08		2.39					
	di-PPH (IV)							2.03		2.10					2.03		2.28		1.98					
	di-PPH		10000	10.0	100.0		2.00	1.93	96.5%	2.21	110%	103.4%	13%	14%	2.09	105%	2.07	104%	2.11	105%	104%	0.81%	0.78%	0.8%
	CH_2Cl_2			950																				
FAMEs Mix (Supelco, Lot# LC00126) [FAME] (mg/mL): 10.0 (Prepared on 6/18/14)	Octanoic acid methyl ester	1.9%	190		19.0		0.95	0.83	87.4%	1.00	105%	96.3%	19%	18%	1.01	106%	1.02	107%	1.01	106%	107%	0.61%	0.57%	0.0%
	Decanoic acid methyl ester	3.2%	320		32.0		1.60	1.35	84.4%	1.65	103%	93.8%	20%	19%	1.64	103%	1.63	102%	1.68	105%	103%	1.7%	1.6%	2.5%
	Dodecanoic acid methyl ester	6.4%	640		64.0		3.20	3.18	99.4%	3.72	116%	107.8%	16%	17%	3.68	115%	3.50	109%	3.66	114%	113%	3.1%	2.7%	-0.6%
	Tridecanoic Acid methyl ester	3.2%	320		32.0		1.60	1.52	95.0%	1.79	112%	103.4%	16%	17%	1.72	108%	1.68	105%	1.76	110%	108%	2.5%	2.3%	2.5%
	Myristoleic Acid methyl ester (C14:1n9c)	1.9%	190		19.0		0.95	0.81	85.3%	1.03	108%	96.8%	24%	23%	0.93	97.9%	0.92	96.8%	1.00	105%	100%	4.6%	4.6%	7.4%
	Tetradecanoic acid methyl ester	3.2%	320		32.0		1.60	1.49	93.1%	1.75	109%	101.3%	16%	16%	1.72	108%	1.65	103%	1.70	106%	106%	2.3%	2.1%	-1.3%
	Pentadecanoic Acid methyl ester	1.9%	190		19.0		0.95	0.85	89.5%	1.02	107%	98.4%	18%	18%	0.93	97.9%	0.93	97.9%	0.98	103%	99.6%	3.0%	3.0%	5.3%
	Palmitoleic Acid methyl ester (C16:1n9c)	6.4%	640		64.0		3.20	2.93	91.6%	3.48	109%	100.2%	17%	17%	3.27	102%	3.21	100%	3.41	107%	103%	3.2%	3.1%	4.4%
	Hexadecanoic acid methyl ester	13.0%	1300		130		6.50	6.23	95.8%	7.15	110%	102.9%	14%	14%	6.80	105%	6.69	103%	7.03	108%	105%	2.7%	2.5%	3.5%
	Heptadecanoic Acid methyl ester	3.2%	320	100.0	32.0	50.0	1.60	1.40	87.5%	1.63	102%	94.7%	15%	14%	1.60	100%	1.56	97.5%	1.64	103%	100%	2.5%	2.5%	2.5%
	Linoleic Acid methyl ester (C18:2n6c)	13.0%	1300		130		6.50	5.80	89.2%	6.71	103%	96.2%	15%	14%	6.50	100%	6.41	98.6%	6.53	100%	99.7%	0.96%	0.96%	0.5%
	Linolenic Acid methyl ester (C18:3n3)- 6.4% Oleic Acid methyl ester (C18:1n9c)- 19.6%	26.0%	2600		260		13.00	12.63	97.2%	13.96	107%	102.3%	10%	10%	13.84	106%	14.03	108%	14.13	109%	108%	1.1%	1.1%	2.2%
	Elaidic Acid methyl ester (C18:1n9t)	2.6%	260		26.0		1.30	1.14	87.7%	1.31	101%	94.2%	14%	13%	1.32	102%	1.33	102%	1.41	108%	104%	3.8%	3.6%	6.9%
	Octadecanoic acid methyl ester	6.5%	650		65.0		3.25	2.93	90.2%	3.31	102%	96.0%	12%	12%	3.36	103%	3.22	99.1%	3.41	105%	102%	3.0%	3.0%	1.5%
	cis-11-Eicosenoic Acid methyl ester (C20:1)	1.9%	190		19.0		0.95	0.74	77.9%	0.91	95.8%	86.8%	21%	18%	0.88	92.6%	0.89	93.7%	0.96	101%	95.8%	4.6%	4.8%	8.4%
	Eicosenoic Acid methyl ester	1.9%	190		19.0		0.95	0.81	85.3%	0.91	95.8%	90.5%	12%	11%	0.91	95.8%	0.89	93.7%	0.96	101%	96.8%	3.8%	3.9%	5.3%
	Erucic Acid methyl ester (C22:1n9)	1.9%	190		19.0		0.95	0.76	80.0%	0.91	95.8%	87.9%	18%	16%	0.86	90.5%	0.86	90.5%	0.87	91.6%	90.9%	0.61%	0.67%	1.1%
	Docosanoic Acid methyl ester	1.9%	190		19.0		0.95	0.77	81.1%	0.93	97.9%	89.5%	19%	17%	0.86	90.5%	0.84	88.4%	0.89	93.7%	90.9%	2.6%	2.9%	3.2%
	CH_2Cl_2			900.0		830.0																		
											min=	80.6%	mean	17%						min=	80.7%		mean	3.4%
											max=	108%	s	5.2%						max=	113%		s	4.1%
													rsd	30%									rsd	123%

[1]- Run Drift = % Target $Value_{last\ CCV}$ - % Target $Value_{first\ CCV}$

[2]- RDP = Relative Percent Difference = abs[$\%target_{second\ CCV}$ - $\%target_{first\ CCV}$]/Average % Target

8-4-14

[redacted]

File: Fatty Acid Sample Analysis.xlsx, Worksheet:CCV Eval

[redacted] 6/30/14

RP1581: Freedom Industries

Continuing Calibration Verification (CCV) Evaluation

Mix	Analyte		Mix Preparation: [Analyte]$_{stk}$ (ug/mL)	Mix Preparation: Stk Sol'n Aliquot (uL)	Mix Preparation: [Analyte]$_{Mix}$ (ug/mL)	CCV Standard Preparation: Mix Aliquot (uL)	CCV Standard Preparation: [Analyte]$_{known}$ (ug/mL)	Matrix Spike #2, Run Date: 6/30/14 — CCV Std Inj #1: [Analyte]$_{obs}$ (ug/mL)	CCV Std Inj #1: % of Target	CCV Std Inj #2: [Analyte]$_{obs}$ (ug/mL)	CCV Std Inj #2: % of Target	CCV Std Inj #3: [Analyte]$_{obs}$ (ug/mL)	CCV Std Inj #3: % of Target	CCV Std Inj #4: [Analyte]$_{obs}$ (ug/mL)	CCV Std Inj #4: % of Target	% of Target$_{n=4}$: mean	% of Target$_{n=4}$: s	% of Target$_{n=4}$: rsd	Run Drift[1]	Overall Results % of Target$_{n=13}$: *mean*	*s*	*rsd*
Fatty Acids (see p.61488)	Octanoic Acid		6477	15.5	100.4	100	10.0	9.74	97.0%	10.89	108%	11.05	110%	12.42	124%	110%	11%	10%	27%	*101%*	*11%*	*11%*
	Decanoic Acid		3330	30.0	99.9		10.0	9.58	95.9%	10.42	104%	11.14	112%	12.11	121%	108%	11%	9.9%	25%	*104%*	*12%*	*12%*
	Dodecanoic Acid		2891	34.5	99.7		10.0	9.76	97.9%	11.12	112%	12.02	121%	12.52	126%	114%	12%	11%	28%	*104%*	*11%*	*11%*
	Tetradecanoic Acid (a.ka. Myristic)		3047	33.0	100.6		10.1	9.48	94.2%	10.63	106%	11.36	113%	11.93	119%	108%	11%	9.7%	24%	*97.8%*	*11%*	*11%*
	Hexadecanoic Acid (a.k.a. Palmitic)		2645	38.0	100.5		10.1	8.18	81.4%	9.24	91.9%	9.69	96.4%	10.49	104%	93.5%	9.6%	10%	23%	*86.8%*	*9.5%*	*11%*
	Linoleic Acid		6011	16.5	99.2		9.92	7.18	72.4%	9.62	97.0%	9.98	101%	10.38	105%	93.6%	15%	16%	32%	*89.5%*	*12%*	*13%*
	Oleic Acid		5201	19.0	98.8		9.88	7.38	74.7%	10.76	109%	9.51	96.3%	9.38	94.9%	93.7%	14%	15%	20%	*92.1%*	*11%*	*11%*
	Octadecanoic Acid (a.k.a. Stearic)		4133	24.0	99.2		9.92	7.69	77.5%	9.10	91.7%	9.01	90.8%	7.38	74.4%	83.6%	8.9%	11%	-3.1%	*87.1%*	*7.6%*	*8.7%*
	Eicosanoic Acid		3843	26.0	99.9		10.0	N.D.	------	N.D.	------	N.D.	------	N.D.	------	-----	-----	-----	-----	-----	-----	-----
	CH_2Cl_2			763.5																		
MCHM Product (*from A. Hunter, see p.60290-60292*)	*MCHM (I)*							*2.04*		*2.36*		*2.40*		*2.60*								
	MCHM (II)							*2.07*		*2.51*		*2.42*		*2.59*								
	Average MCHM		10000	10.0	100.0	20.0	2.00	2.06	103%	2.44	122%	2.41	121%	2.60	130%	119%	11%	9.6%	27%	*110%*	*11%*	*10%*
	PPH		10000	10.0	100.0		2.00	2.21	111%	2.37	119%	2.40	120%	2.58	129%	120%	7.6%	6.3%	19%	*112%*	*10%*	*9.3%*
	1,4-CHDM (I)							*1.98*		*2.44*		*2.80*		*2.91*								
	1,4-CHDM (II)							*2.19*		*2.21*		*2.44*		*2.68*								
	1,4-CHDM		10000	10.0	100.0		2.00	2.09	104%	2.33	116%	2.62	131%	2.80	140%	123%	16%	13%	36%	*115%*	*15%*	*13%*
	DM-1,4-DC		10000	10.0	100.0		2.00	2.22	111%	2.38	119%	2.57	129%	2.66	133%	123%	9.8%	8.0%	22%	*115%*	*10%*	*8.9%*
	di-PPH (I)							*1.97*		*2.24*		*2.41*		*2.63*								
	di-PPH (II)							*2.17*		*2.45*		*2.55*		*2.73*								
	di-PPH (III)							*2.57*		*2.51*		*1.90*		*2.13*								
	di-PPH (IV)							*2.19*		*2.48*		*1.85*		*1.73*								
	di-PPH		10000	10.0	100.0		2.00	2.23	111%	2.42	121%	2.18	109%	2.31	115%	114%	5.3%	4.6%	4.0%	*110%*	*7.1%*	*6.4%*
	CH_2Cl_2			950																		
FAMEs Mix (Supelco, Lot# LC00126) [FAME] (mg/mL): 10.0 (Prepared on 6/18/14)	Octanoic acid methyl ester	1.9%	190	100.0	19.0	50.0	0.95	1.03	108%	1.17	123%	1.14	120%	1.19	125%	119%	7.5%	6.3%	17%	*111%*	*10%*	*9.1%*
	Decanoic acid methyl ester	3.2%	320		32.0		1.60	1.68	105%	1.71	107%	1.90	119%	1.99	124%	114%	9.3%	8.2%	19%	*108%*	*10%*	*9.5%*
	Dodecanoic acid methyl ester	6.4%	640		64.0		3.20	3.84	120%	4.08	128%	4.30	134%	4.42	138%	130%	8.0%	6.1%	18%	*119%*	*10%*	*8.8%*
	Tridecanoic Acid methyl ester	3.2%	320		32.0		1.60	1.82	114%	2.02	126%	2.16	135%	2.17	136%	128%	10%	8.0%	22%	*115%*	*12%*	*10%*
	Myristoleic Acid methyl ester (C14:1n9c)	1.9%	190		19.0		0.95	1.01	106%	1.09	115%	1.19	125%	1.19	125%	118%	9.2%	7.8%	19%	*108%*	*11%*	*10%*
	Tetradecanoic acid methyl ester	3.2%	320		32.0		1.60	1.84	115%	2.00	125%	2.11	132%	2.11	132%	126%	8.0%	6.3%	17%	*114%*	*11%*	*9.9%*
	Pentadecanoic Acid methyl ester	1.9%	190		19.0		0.95	1.05	111%	1.12	118%	1.19	125%	1.24	131%	121%	8.7%	7.2%	20%	*109%*	*11%*	*11%*
	Palmitoleic Acid methyl ester (C16:1n9c)	6.4%	640		64.0		3.20	3.57	112%	3.86	121%	4.09	128%	4.09	128%	122%	7.7%	6.3%	16%	*110%*	*11%*	*9.6%*
	Hexadecanoic acid methyl ester	13.0%	1300		130		6.50	7.58	117%	8.12	125%	8.55	132%	8.55	132%	126%	7.1%	5.6%	15%	*113%*	*11%*	*9.5%*
	Heptadecanoic Acid methyl ester	3.2%	320		32.0		1.60	1.75	109%	1.89	118%	2.04	128%	2.06	129%	121%	9.0%	7.5%	19%	*108%*	*12%*	*11%*
	Linoleic Acid methyl ester (C18:2n6c)	13.0%	1300		130		6.50	6.98	107%	7.61	117%	8.24	127%	8.24	127%	120%	9.3%	7.8%	19%	*107%*	*11%*	*10%*
	Linolenic Acid methyl ester (C18:3n3)- 6.4% Oleic Acid methyl ester (C18:1n9c)- 19.6%	26.0%	2600		260		13.00	14.97	115%	15.80	122%	16.83	129%	16.80	129%	124%	6.9%	5.5%	14%	*113%*	*9.2%*	*8.1%*
	Elaidic Acid methyl ester (C18:1n9t)	2.6%	260		26.0		1.30	1.41	108%	1.50	115%	1.58	122%	1.50	115%	115%	5.3%	4.6%	6.9%	*107%*	*8.7%*	*8.2%*
	Octadecanoic acid methyl ester	6.5%	650		65.0		3.25	3.64	112%	3.88	119%	4.16	128%	4.13	127%	122%	7.5%	6.2%	15%	*109%*	*11%*	*9.9%*
	cis-11-Eicosenoic Acid methyl ester (C20:1)	1.9%	190		19.0		0.95	0.97	102%	1.04	109%	0.99	104%	1.05	111%	107%	4.1%	3.8%	8.4%	*100%*	*8.5%*	*8.6%*
	Eicosenoic Acid methyl ester	1.9%	190		19.0		0.95	0.98	103%	1.05	111%	1.13	119%	1.13	119%	113%	7.6%	6.7%	16%	*102%*	*9.7%*	*9.5%*
	Erucic Acid methyl ester (C22:1n9)	1.9%	190		19.0		0.95	0.87	91.6%	1.00	105%	1.08	114%	1.11	117%	107%	11%	11%	25%	*97.4%*	*11%*	*11%*
	Docosanoic Acid methyl ester	1.9%	190		19.0		0.95	0.92	96.8%	1.03	108%	1.11	117%	1.13	119%	110%	10%	9.1%	22%	*99.8%*	*11%*	*11%*
	CH_2Cl_2			900.0		830.0																

min= 83.6%	mean 19%	*86.8% = min*	
max= 130%	s 7.9%	*119% = max*	
	rsd 41%		

1- Run Drift = % Target Value$_{last\ CCV}$ - % Target Value$_{first\ CCV}$

2- RDP = Relative Percent Difference = abs[%target$_{second\ CCV}$ - %target$_{first\ CCV}$]/Average % Target

8-1-14 [redacted]

RP1581: Freedom Industries

	Replicate Sample Preparations											
	Tank 393, L-1				Tank 203, L-1				Tank 203, S-2			
Analyte	$mean_{Content (\%)}$	s	rsd	n	$mean_{Content (\%)}$	s	rsd	n	$mean_{Content (\%)}$	s	rsd	n
Octanoic Acid	1.88%	0.13%	6.7%	3	1.17%	0.048%	4.1%	3	1.30%	0.044%	3.4%	3
Decanoic Acid	1.12%	------	------	2	<R.L.	------	------	------	<R.L.	------	------	------
Dodecanoic Acid	1.99%	0.14%	7.0%	3	1.29%	0.073%	5.7%	3	1.44%	0.072%	5.0%	3
Tetradecanoic Acid	1.80%	0.091%	5.1%	3	1.54%	0.11%	7.4%	3	1.81%	0.085%	4.7%	3
Hexadecanoic Acid	2.46%	0.12%	4.9%	3	2.72%	0.12%	4.5%	3	5.34%	0.54%	10%	3
Linoleic Acid	5.90%	1.2%	20%	3	9.70%	1.1%	11%	3	9.83%	0.49%	5.0%	3
Oleic Acid	5.80%	1.0%	17%	3	6.51%	0.67%	10%	3	6.21%	0.44%	7.1%	3
Octadecanoic Acid	<R.L.	------	------	------	<R.L.	------	------	------	<R.L.	------	------	------
Eicosanoic Acid	<R.L.	------	------	------	<R.L.	------	------	------	<R.L.	------	------	------
Average MCHM[a]	0.246%	0.014%	5.8%	3	0.413%	0.0046%	1.1%	3	0.377%	0.018%	4.9%	3
PPH	<R.L.	------	------	------	<R.L.	------	------	------	<R.L.	------	------	------
Average 1,4-CHDM	<R.L.	------	------	------	<R.L.	------	------	------	<R.L.	------	------	------
DM-1,4-DC	<R.L.	------	------	------	<R.L.	------	------	------	<R.L.	------	------	------
Average di-PPH[a]	0.214%	0.039%	18%	3	0.770%	0.084%	11%	3	0.676%	0.070%	10%	3
Octanoic acid methyl ester	0.110%	0.0046%	4.1%	3	0.0499%	0.0026%	5.2%	3	<R.L.	------	------	------
Decanoic acid methyl ester	<R.L.	------	------	------	<R.L.	------	------	------	<R.L.	------	------	------
Dodecanoic acid methyl ester	<R.L.	------	------	------	<R.L.	------	------	------	<R.L.	------	------	------
Tridecanoic Acid methyl ester	<R.L.	------	------	------	<R.L.	------	------	------	<R.L.	------	------	------
Myristoleic Acid methyl ester (C14:1n9c)	0.0650%	0.0034%	5.2%	3	<R.L.	------	------	------	<R.L.	------	------	------
Tetradecanoic acid methyl ester	0.199%	0.012%	5.9%	3	0.0980%	0.0027%	2.8%	3	0.0979%	0.0018%	1.9%	3
Pentadecanoic Acid methyl ester	0.0548%	------	------	2	<R.L.	------	------	------	<R.L.	------	------	------
Palmitoleic Acid methyl ester (C16:1n9c)	0.511%	0.026%	5.0%	3	0.236%	0.0088%	3.7%	3	0.242%	0.021%	8.7%	3
Hexadecanoic acid methyl ester	4.15%	0.043%	1.0%	3	2.69%	0.052%	1.9%	3	2.39%	0.018%	0.77%	3
Heptadecanoic Acid methyl ester	0.0975%	0.0069%	7.1%	3	<R.L.	------	------	------	<R.L.	------	------	------
Linoleic Acid methyl ester (C18:2n6c)	9.76%	0.17%	1.7%	3	9.31%	0.12%	1.3%	3	8.42%	0.13%	1.5%	3
Linolenic Acid methyl ester (C18:3n3) Oleic Acid methyl ester (C18:1n9c)	12.75%	0.11%	0.87%	3	7.44%	0.21%	2.9%	3	6.77%	0.056%	0.83%	3
Elaidic Acid methyl ester (C18:1n9t)	0.874%	0.089%	10%	3	0.405%	0.019%	4.8%	3	0.343%	0.022%	6.5%	3
Octadecanoic acid methyl ester	1.54%	0.016%	1.0%	3	0.914%	0.019%	2.0%	3	0.834%	0.020%	2.4%	3
cis-11-Eicosenoic Acid methyl ester (C20:1)	0.179%	0.013%	7.2%	3	0.125%	0.0087%	7.0%	3	0.129%	0.0043%	3.3%	3
Eicosanoic Acid methyl ester	0.119%	0.0024%	2.0%	3	0.0919%	0.0031%	3.3%	3	0.0961%	0.0049%	5.1%	3
Erucic Acid methyl ester (C22:1n9)	<R.L.	------	------		<R.L.	------	------	------	<R.L.	------	------	------
Docosanoic Acid methyl ester	0.119%	0.0062%	5.2%	3	0.106%	0.0040%	3.8%	3	0.116%	0.0055%	4.7%	3
min =			0.87%				1.1%				0.77%	
max=			20%				11%				10%	

RP1581: Freedom Industries

Analyte	QC Events									
	CCV				SS				MS Samples: Average Recovery	
	mean% Target	s	rsd	n	mean% Target	s	rsd	n	Tank 393, L-1	Tank 203, S-2
Octanoic Acid	101%	11%	11%	13	102%	0.71%	0.69%	3	90.7%	------
Decanoic Acid	104%	12%	12%	13	105%	1.8%	1.7%	3	88.4%	------
Dodecanoic Acid	104%	11%	11%	13	102%	1.2%	1.2%	3	89.4%	80.3%
Tetradecanoic Acid	97.8%	11%	11%	13	95.8%	0.90%	0.94%	3	63.4%	70.1%
Hexadecanoic Acid	86.8%	9.5%	11%	13	82.2%	1.1%	1.3%	3	53.4%	112%
Linoleic Acid	89.5%	12%	13%	13	82.0%	1.3%	1.6%	3	85.3%	------
Oleic Acid	92.1%	11%	11%	13	85.2%	2.5%	2.9%	3	------	106%
Octadecanoic Acid	87.1%	7.6%	8.7%	13	84.8%	3.3%	3.9%	3	------	------
Eicosanoic Acid	-----	-----	-----	13	-----	-----	-----	3	------	------
Average MCHM[a]	110%	11%	10%	13	112%	0.66%	0.59%	3	117%	119%
PPH	112%	10%	9.3%	13	102%	2.2%	2.1%	3	------	------
Average 1,4-CHDM	115%	15%	13%	13	101%	8.4%	8.3%	3	------	------
DM-1,4-DC	115%	10%	8.9%	13	113%	1.1%	0.94%	3	------	------
Average di-PPH[a]	110%	7.1%	6.4%	13	112%	9.3%	8.3%	3	124%	124%
Octanoic acid methyl ester	111%	10%	9.1%	13	102%	1.8%	1.8%	3	110%	------
Decanoic acid methyl ester	108%	10%	9.5%	13	100%	2.2%	2.2%	3	------	------
Dodecanoic acid methyl ester	119%	10%	8.8%	13	107%	2.8%	2.6%	3	------	------
Tridecanoic Acid methyl ester	115%	12%	10%	13	103%	3.3%	3.2%	3	------	------
Myristoleic Acid methyl ester (C14:1n9c)	108%	11%	10%	13	98.2%	0.61%	0.62%	3	------	------
Tetradecanoic acid methyl ester	114%	11%	9.9%	13	101%	0.36%	0.36%	3	115%	------
Pentadecanoic Acid methyl ester	109%	11%	11%	13	97.5%	1.6%	1.6%	3	------	------
Palmitoleic Acid methyl ester (C16:1n9c)	110%	11%	9.6%	13	99.2%	1.0%	1.0%	3	113%	------
Hexadecanoic acid methyl ester	113%	11%	9.5%	13	99.6%	0.62%	0.62%	3	119%	126%
Heptadecanoic Acid methyl ester	108%	12%	11%	13	94.4%	0.62%	0.66%	3	------	------
Linoleic Acid methyl ester (C18:2n6c)	107%	11%	10%	13	96.1%	1.4%	1.5%	3	------	------
Linolenic Acid methyl ester (C18:3n3) Oleic Acid methyl ester (C18:1n9c)	113%	9.2%	8.1%	13	101%	1.0%	1.0%	3	109%	115%
Elaidic Acid methyl ester (C18:1n9t)	107%	8.7%	8.2%	13	97.9%	2.2%	2.3%	3	103%	118%
Octadecanoic acid methyl ester	109%	11%	9.9%	13	96.3%	1.9%	1.9%	3	116%	119%
cis-11-Eicosenoic Acid methyl ester (C20:1)	99.7%	8.5%	8.6%	13	90.5%	2.8%	3.1%	3	88.9%	93.3%
Eicosanoic Acid methyl ester	102%	9.7%	9.5%	13	90.2%	1.6%	1.8%	3	100%	------
Erucic Acid methyl ester (C22:1n9)	97.4%	11%	11%	13	87.4%	0.0%	0.0%	3	------	------
Docosanoic Acid methyl ester	100%	11%	11%	13	87.7%	1.6%	1.8%	3	97.6%	104%
min =										
max=										

Overall Recovery	
mean=	102%
s=	19%
rsd=	18%
n=	30
min=	53.4%
max=	126%

Name	Project No.	Location	61487
[redacted]	RP1581		Date: 6/18/14

Sample Analysis

• Fatty Acid Mix: The following stock solution aliquots were combined with CH_2Cl_2 to produce a final solution of 1000 µl.

Stock Soln	Aliquot (µl)	$[Analyte]_{Stk}$ ($\frac{\mu g}{ml}$)	* $[Analyte]_{soln}$ ($\frac{\mu g}{ml}$)
Octanoic Acid	15.5	6477 ✓	100.4 ✓
Decanoic Acid	30.0	3330 ✓	99.90 ✓
Dodecanoic Acid	34.5	2891 ✓	99.74 ✓
Myristic Acid	33.0	3047 ✓	100.6 ✓
Palmitic Acid	38.0	2645 ✓	100.5 ✓
Stearic Acid	24.0	4133 ✓	99.19 ✓
Eicosanoic Acid	26.0	3843 ✓	99.92 ✓
Linoleic Acid	16.5	6011 ✓	99.18 ✓
Oleic Acid	19.0	5201 ✓	98.82 ✓
CH_2Cl_2	763.5 ✓	—	—
Total Volume (µl)	1000 ✓		

$$^{*}[Analyte]_{soln}\left(\frac{\mu g}{ml}\right) = \frac{[Analyte]_{Stk}\left(\frac{\mu g}{ml}\right) \times Aliquot\,(\mu l)}{1000\,\mu l} \checkmark$$

• MCNM Mix: Diluted 100 µl of 1000 $\frac{\mu g}{ml}$ MCNM mix from A. Hunter ✓ (prep on 5/12/14) with 900 µl of ✓ CH_2Cl_2. $[Analyte]_{soln} = \frac{1000 \frac{\mu g}{ml} \times 100\,\mu l}{1000\,\mu l} = 100 \frac{\mu g}{ml}$ ✓

• FAME Mix: Diluted 100 µl of the 10,000 $\frac{\mu g}{ml}$ FAME mix with 900 µl of CH_2Cl_2. ~~[Analyte]~~ [Solution] = 1000 $\frac{\mu g}{ml}$ ✓ 6/18/14

Calibration Solutions: Aliquots from the three mixes were combined w/ CH_2Cl_2 to produce the following calibration solns.

Aliquots Used (µl)

Mix	Std 1	Std 2	Std 3	Std 4	Std 5	Std 6	
100 Fatty Acid ✓	20.0	50.0	75.0	100	200	400	Total Volume for
100 MCNM ✓	2.00	5.00	10.0	20.0	50.0	100	all Std solns:
1000 FAME ✓	5.00	10.0	25.0	50.0	75.0	150	1000 µl
CH_2Cl_2	973	935	890	830	675	350	7-29-14 [redacted]

Name	Project No. Location	61488
[redacted]	RP1581	Date: 6/18/14

Sample Analysis:

- The six calibration solutions along with CH_2Cl_2 blanks and a 50 ppm DFTPP solution were injected into the GC/MS system.

6/19/14

All six calibration solutions, along with the DFTPP solution were injected into the GC/MS. This data was used to set up the calibration curve for each analyte.

6/20/14

Prepared another 100 ppm fatty acid mix since the nearly all of the solution prepared on 6/18/14 was used in the calibration solution preparation. Used ~~sample~~ mix DW 6/20/14 prep process shown on p. 61487. Also prepared a Std 4 CCV standard using this fatty acid mix along with the MCAM + FAME mixes prepared on 6/18/14, also see p. 61487.

Balance: # 4338

✓ wt set: EPA-K-5
100 mg => 0.1000 g
1 g => 1.0000 g
20 g => 20.0004 g

The following sample aliquots were placed in tared, ~~4 mL~~ 12 DW 6/20/14 clear glass vials. Samples were removed from walk-in cooler ~~prior~~ prior DW 6/20/14 to the aliquot removal.

Sample	Tag #	Phase	~~Sample~~ Aliquot Amount (g)	Replicate
Tank 393	H01500	L-1	0.1135	A
"	"	"	0.1148	B
"	"	"	0.1045	C
Tank 203	none	L-1	0.1263	A
"	"	"	0.1401	B
"	"	"	0.1195	C
"	"	S-2	0.1127	A
"	"	"	0.1028	B
"	"	"	0.1095	C

Name	Project No.	Location	61489
[redacted]	RP1581		Date: 6/20/14

Sample Analysis

- Into each vial, 6.00 ml of a 30% THF in IPA solution was added using a type-A glass pipet, see p. 61488 for ~~extraction~~ 30% THF in IPA DH. 6/20/14 soln prep.

- An extraction blank was also prepared by placing 6.00 ml of the THF/IPA soln in an empty ~~8~~ 12 ml clear glass vial. DH. 6/20/14

Each vial was:
- vortexed mixed for 15 seconds
- sonicated for 5 mins
- centrifuged @ 1500 rpm for 5 mins.

For each liquid layer, a 10.0 µl aliquot was combined with 990 µl of CH_2Cl_2, producing a 100x dilution of the extract.

For the tank 203, S-2 samples, ~~scald~~ solid DH, 6/20/14 material was present on the vial bottoms after centrifugation.

Second Source: The following stock solutions were prepared and used in the preparation of a second source (SS) standard solution. These stocks were prepared from the neat materials listed on p. 61468 and 61479.

Acid	Purity (%)	Amount in tared vial (g)
Linoleic	99.9	0.0307 0.0302 DH 6/20/14
Oleic	99.7	0.0277
Octanoic	99.9	0.0366
Decanoic	99.9	0.0598
Dodecanoic	99.7	0.0358
Myristic	99.9	0.0302
Palmitic	99.8	0.0390
Stearic	99.6	0.0331
Eicosanoic	99.4	0.0379

Prepared more 30% THF in IPA soln by combining 30 ml of THF (BHJ, Lot # DA191) and 70 ml of IPA (Fisher, Lot # 132649)

Added 1.00 ml of the THF/IPA soln to each vial which was then vortexed mixed and sonicated for 5 min.

7-29-14

Name	Project No.	Location	61490
[redacted]	RP1581		Date: 6/20/14

Sample Analysis

$[\text{Linoleic}]_{ss\ stk} = \frac{0.0302\,g \times 0.999}{6.00\,mL} \times \frac{1\times10^6\,\mu g}{g} = 5028\ \frac{\mu g}{mL}$

$[\text{Oleic}]_{ss\ stk} = \frac{0.0277\,g \times 0.997}{6.00\,mL} \times \frac{1\times10^6\,\mu g}{g} = 4603\ \frac{\mu g}{mL}$

$[\text{Octanoic}]_{ss\ stk} = \frac{0.0366 \times 0.999}{6.00\,mL} \times \frac{1\times10^6\,\mu g}{g} = 6094\ \frac{\mu g}{mL}$

$[\text{Decanoic}]_{ss\ stk} = \frac{0.0598 \times 0.999}{6.00\,mL} \times \frac{1\times10^6\,\mu g}{g} = 9957\ \frac{\mu g}{mL}$

$[\text{Dodecanoic}]_{ss\ stk} = \frac{0.0358 \times 0.997}{6.00\,mL} \times \frac{1\times10^6\,\mu g}{g} = 5949\ \frac{\mu g}{mL}$

$[\text{Myristic}]_{ss\ stk} = \frac{0.0302 \times 0.999}{6.00\,mL} \times \frac{1\times10^6\,\mu g}{g} = 5028\ \frac{\mu g}{mL}$

$[\text{Palmitic}]_{ss\ stk} = \frac{0.0390 \times 0.998}{6.00\,mL} \times \frac{1\times10^6\,\mu g}{g} = 6487\ \frac{\mu g}{mL}$

$[\text{Stearic}]_{ss\ stk} = \frac{0.0331 \times 0.996}{6.00\,mL} \times \frac{1\times10^6\,\mu g}{g} = 5495\ \frac{\mu g}{mL}$

$[\text{Eicosanoic}]_{ss\ stk} = \frac{0.0379 \times 0.994}{6.00\,mL} \times \frac{1\times10^6\,\mu g}{g} = 6279\ \frac{\mu g}{mL}$

For the MCHM Products, the following stock solutions were used

$[\text{MCHM}]_{stk} = 9945\ \frac{\mu g}{mL}$
$[\text{PPH}]_{stk} = 9932\ \frac{\mu g}{mL}$
$[\text{1,4-CHDM}] = 10{,}000\ \frac{\mu g}{mL}$
$[\text{DMCH-1,4-DC}] = 9948\ \frac{\mu g}{mL}$

} See p. 61467

$[\text{di-PPH}] = 10{,}000\ \frac{\mu g}{mL}$ - See p. 61468

For the FAMEs, used the FAME mix from Supelco

7-29-14 [redacted]

Name	Project No.	Location	61491
(b) (6), (b) (7)(C) [redacted]	RP1581		Date: 6/20/14

Sample Analysis

SS Fatty Acid Mix:

Acid Analyte	[Stk soln] $\frac{\mu g}{mL}$	Aliquot (µL)	[Analyte]$_{mix}$ ($\frac{\mu g}{mL}$)
Linoleic	5028 ✓	20.0	101 ✓
Oleic	4603 ✓	21.5	99.0 ✓
Octanoic	6094 ✓	16.5	101 ✓
Decanoic	9957 ✓	10.0	99.6 ✓
Dodecanoic	5949 ✓	17.0	101 ✓
Myristic	5028 ✓	20.0	101 ✓
Palmitic	6487 ✓	15.5	101 ✓
Stearic	5495 ✓	18.0	98.9 ✓
Eicosanoic	6279 ✓	16.0	100 ✓
CH_2Cl_2	—	845.5 ✓	—
	Total	1000.0 ✓	

SS MCHM Mix:

Analyte	[Stk soln] $\frac{\mu g}{mL}$	Aliquot (µL)	[Analyte]$_{mix}$ ($\frac{\mu g}{mL}$)
MCHM	9945 ✓	10.0	99.5 ✓
PPh	9932 ✓	10.0	99.3 ✓
1,4-CHDM	10,000 ✓	10.0	100 ✓
DMCH-1,4-DC	9948 ✓	10.0	99.5 ✓
di-PPH	10,000 ✓	10.0	100 ✓
30% THF in IPA	—	950.0 ✓	—
	Total	1000.0 ✓	

Used 30% THF/IPA soln since ~~MCHM~~ these analyte stock soln's were prepared in MeOH. DH, 6/20/14

SS FAME Mix:

Combined 100 µL of the 10,000 ✓ $\frac{\mu g}{mL}$ FAME mix with 900 µL of CH_2Cl_2. FAME mix, Supelco, Lot # LC00126 ✓

SS Std:

Combined: 100 µL of the SS Fatty Acid mix
20 µL of the SS MCHM mix
50 µL of the SS FAME mix
830 µL of CH_2Cl_2

7-29-14 (b) (6), (b) (7) [redacted]

Name	Project No.	Location	61492
[redacted]	RP1581		Date: 6/20/14

Sample Analysis

The CCV and SS standards along with the diluted sample extracts were placed in the GC/MS system for analysis.

6/23/14

Data from the 6/20/14 run was processed on 6/21/14, and based on these results, two matrix spike (MS) samples were prepared.

Balance: # 4338

✓ wt set: EPA-K-5
100 mg => 0.1000g
1 g => 1.0000g
20g => 20.0004g

Into tared 5-mL clear glass vials, the following matrix spike samples were prepared.

Analyte	Amount added (g)
Tank 393, L-1	0.1087
Octanoic Acid	0.0086
Decanoic Acid	0.0054
Dodecanoic Acid	0.0052
Myristic Acid	0.0024
Palmitic Acid	0.0033
Linoleic Acid	0.0088

Analyte	Amount added (g)
Tank 203, S-2	0.1052
Octanoic Acid	0.0073
Dodecanoic Acid	0.0033
Myristic Acid	0.0054
Palmitic Acid	0.0065
Oleic Acid	0.0115

Vials were placed in locked cart and stored in walk-in cooler "B" in room 1C-210.

6/24/14

$[MCHM]_{stk}$ = 15,900 µg/mL ✓ (see p. 60290)
$[di\text{-}PPH]_{stk}$ = 17,650 ✓ µg/mL (see p. 60291)
Aliquots from both stock solutions were added to each MS sample.

MS Sample	Aliquot (µL) MCHM	Aliquot (µL) di-PPH
Tank 393, L-1	17.0	~~26.0~~ 13.0 PH 6/24/14
Tank 203, S-2	~~13.0~~ 26.0 PH 6/24/14	50.0

Name	Project No.	Location	61493
[redacted]	RP1581		Date: 6/24/14

Sample Analysis

- 6.00 ml of a 30% THF in IPA was added to each MS vial and these samples were extracted using the process on p. 61489.

For each MS, 10.0 µl of the resulting extract was combined with 50.0 µl of the 1000 ppm FAME mix (p. 61487) and 940 µl of CH_2Cl_2. The resulting solutions ~~was~~ were injected into the GC/MS system.

A new CCV standard was prepared (see p. 61488) and analyzed.

6/25/14

Decided to prepare another set of MCHM stocks since the sol'ns that were initially used for the second source evaluation were prepared in MeOH rather than CH_2Cl_2.

Balance: # 4338

√ wt set: EPA-K-1
100 mg => 0.1000 g
1 g => 1.0000 g
20 g => 20.0004 g

Into tared, 8 ml amber vials, the following neat materials were placed:

Analyte	Source	Lot #	Purity %	Amount (g)	[Analyte] µg/ml
MCHM	TCI	RCGNA-PT	98.0	0.0406	6631 ✓
PPH	Dow	2Iϕ5ϕ1G3A6	99.8	0.0502	8350 ✓
di-PPH	Dow	20060920-14	100	0.0516	8600 ✓
DMCH-1,4-DC	Aldrich	MKBJ2148V	97	0.0580	9377 ✓
1,4-CHDM	Aldrich	MKBP6651V	99	0.0420	6930 ✓
CH_2Cl_2	EM Science	39194	—	—	—

4.00 ml of CH_2Cl_2 was added to each vial using a type-A volumetric pipet.

7-29-14 [redacted]

Name	Project No.	Location	61494
[redacted]	RP1581		Date: 6/25/14

Sample Analysis

SS MCHM Mix #2

Analyte	[ ]std (µg/ml)	Aliquot (µl)	[ ]mix #2 µg/ml
MCHM	6631 ✓	15.0	~~99.48~~ 99.40 DR, 8/5/14
PPH	8350 ✓	12.0	100.62 ✓
DM-1,4-DC	9377 ✓	10.5	DR 6/25/14 ~~98.5~~ 98.46 ✓
1,4-CHDM	6930 ✓	14.5	100.5 ✓
di-PPH	8600 ✓	11.5	98.90 ✓
CH_2Cl_2	—	936.5 ✓	—
	Total	1000.0 ✓	

SS Std #2:
Combined 20.0 µl of SS MCHM Mix #2 with 100 µl SS FA Mix and 50.0 µl of SS FAME Mix and 830 µl of CH_2Cl_2. Resulting soln was injected into the GC/MS system.

CCV Std:
Prepared a CCV std by combining 20.0 µl of the MCHM Mix (see p. 61487), 50.0 µl of the FAME Mix (see p. 61487) and 100 µl of the FA Mix (see p. 61487) and 830 µl of CH_2Cl_2. Resulting soln was injected into the GC/MS system.

6/30/14

Decided to prep another set of matrix spike solutions ~~samples~~ from the two extracts prepared and extracted on 6/23-24/14, see p. 61492. Extracts were removed from the refrigerator and allowed to warm to ambient temperature. Each vial was vortexed mixed for about 10 seconds, then centrifuged for 5 min at 1500 rpm. A 10 µl extract aliquot from each vial was combined with 50 µl of the 1000 ppm FAME mix and 940 µl of CH_2Cl_2. Each matrix spike solution was injected three times.

7-29-14 [redacted]

Name	Project No.	Location	61495
[redacted]	RM581		Date: 6/30/14

Sample Analysis

Prepared:

- A 50 ppm DFTPP in CH_2Cl_2 by combining 50 µl of a 1000 ppm DFTPP soln (Supelco, Lot #LB91234) with 950 µl of CH_2Cl_2

- A CCV standard by combining
  - 100 µl of the 100 ppm FA ~~soln~~ mix [initials] 6/30/14
  - 20 µl of the 100 ppm MCHM mix
  - 50 µl of the 1000 ppm FAME mix
  - 830 µl of CH_2Cl_2

End 7/24/14

[redacted]

7-29-14 [redacted]

Name	Project No.	Location	61466
[redacted]	RP1581		Date: 4/9/14

Standard Preparations

Balance: #4948

✓ wt set: EPA-K-~~3~~ 5 DH 4/9/14
20 mg => 0.0200g
100 mg => 0.1000g
1g => 1.0000g
20g => 20.0003g

Acid Standard	Lot #	Purity	Amount placed in tared vial (g)
Linoleic,	Aldrich - BCBD3934V:	60-~~70~~ 74% DH 4/9/14	0.0372
Isovaleric,	Aldrich - 24507AB:	99%	0.0465
Oleic,	Aldrich - MKBG6690V:	90%	0.0407
Propionic,	Aldrich - 11318JA:	99.5%	0.0471
Butyric,	Aldrich - 03511DA:	99%	0.0503
Octanoic,	Aldrich - 14525PA:	99.5%	0.0513
Hexanoic,	Aldrich - 01512ES,	99.5%	0.0504
①Palmitic,	Sigma - 091M1437V,	99%	0.0420
①Stearic,	Sigma - 15303DE,	95%	0.0410

~~Lotto~~ DH 4/9/14

Other standards:

①1,4-Cyclohexanedimethanol, Aldrich - MKBP6651V: 99% - 0.0480g
(1,4-CH-di-MeOH)

Dimethyl cyclohexane-1,4-dicarboxylate, Aldrich - MKBJ2148V - 0.0441g, 97%
(DMCH-1,4-DC)

1-Phenoxy-2-propanol, Aldrich - MKBK0424V - 0.0534g, 93%
(PPH)

Crude PPH, Received from J. Gundersen, 4/2/14 - 0.0484g

Crude MCHM, Received from J. Gundersen, 4/2/14 - 0.0458g

① - solid material, all other compounds were liquids.

4/10/14

Balance: #4338

✓ wt set: EPA-K-5
20 mg => 0.0200g
100 mg => 0.1000g
1g => 1.0000g
20g => 20.0002g

4-methyl-1-cyclohexanemethanol (MCHM)
TCI, Lot # RCGNA-PT, 98%
amount in tared vial: 0.0548g

Name	Project No.	Location	61467
[redacted]	RP1581		Date: 4/10/14

Standard Preparations

MeOH (Fisher, Lot # 128149) was added to each vial, except for the isovaleric, stearic and palmitic acids. For these standards 6 ml of a diluent (DN, 4/10/14) was added that included 3 ml of MeOH and 3 ml of acetone. These solutions were then sonicated in a water bath for 10 min.

Stock solution concentrations:

Analyte	Amount (g)	Purity	Diluent Volume (mL)	[Analyte] µg/mL
linoleic acid	0.0372	60 %	4.460	5004 ✓
isovaleric acid	0.0465	99 %	6.000 *	7673
oleic acid	0.0407	90 %	3.600	10180 ✓
propionic acid	0.0471	99.5 %	4.700	9971
butyric acid	0.0503	99 %	5.000	9959
DN 4/10/14 ~~Octanic~~ octanoic acid	0.0513	99.5 %	5.100	10010
hexanoic acid	0.0504	99.5 %	5.000	10030
palmitic acid	0.0420	99 %	6.000 *	6930
stearic acid	0.0410	95 %	6.000 *	6492
1,4-CH-di-MeOH	0.0480	99 %	4.750	10000 ✓
DMCH-1,4-DC	0.0441	97 %	4.300	9948 ✓
PPH	0.0534	93 %	5.000	9932
MCHM	0.0548	98 %	5.400	9945 ✓
Crude PPH	0.0484	—	4.800	10080
Crude MCHM	0.0458	—	4.600	9957

** - diluent was 3 ml MeOH + 3 ml acetone

- $[\text{Analyte}] = \dfrac{\text{Amount (g)} \times \text{Purity}}{\text{Diluent Volume (mL)}} \times \dfrac{1\times10^6\ \mu g}{g}$

$$[\text{PPH}] = \frac{0.0534\ g \times 0.93}{5.000\ mL_{\text{diluent}}} \times \frac{1\times10^6\ \mu g}{g} = 9932\ \frac{\mu g}{mL}$$

- For Crude PPH + Crude MCHM, purity was assumed to be 100 %.

- Glycerol, Sigma, Lot # 00157AH, 99.5 %
  amount placed in tared amber glass vial: 0.0655 g
  Diluent volume: 6.500 mL

$$[\text{Glycerol}] = \frac{0.0655\ g \times 0.995}{6.500\ mL\ \text{diluent}} \times \frac{1\times10^6\ \mu g}{g} = 10030\ \frac{\mu g}{mL}$$

[redacted]
7-29-14

Name	Project No.	Location	61468
[redacted]	RP1581		Date: 5/2/14

Standard Prep

- Balance: # 4338

✓ wt set: EPA-K-5
20 mg => 0.0200 g
1 g => 1.0000 g
20 g => 20.0001 g

- Dipropylene Glycol Phenyl Ether (DiPPh)
  Dow Chemical, Lot # 200602920-14.
  Purity: 100% (assumed)
  Amount placed in a tared 7 mL amber vial: 0.0596 g

- Cyclohexanol, Sigma-Aldrich, Lot # 11107C1?, purity ≥ 99%
  Amount placed in a tared 7-mL amber vial: 0.0644 g

- MeOH, Fisher, Lot # 128149

$$[\text{DiPPh}] = \frac{0.0596\,g}{5.960\,mL_{MeOH}} \times \frac{1\times10^6\,\mu g}{g} = \underline{10,000}\ \frac{\mu g}{mL}$$

$$[\text{Cyclohexanol}] = \frac{0.0644\,g}{6.640\,mL_{MeOH}} \times 0.99 \times \frac{1\times10^6\,\mu g}{g} = \underline{9602}\ \frac{\mu g}{mL}$$

MeOH volumes (6.00 mL) dispensed using Type-A glass pipets and 1000 µL Hamilton syringe.

5/19/14

Balance: # 4338

✓ wt set: EPA-K-5
20 mg => 0.0200 g
1 g => 1.0001 g
20 g => 20.0002 g

Fatty Acids Kit, Supelco, Lot # LC06366
The following neat materials were placed in a tared 8-mL amber glass vials:

Acid Compound	Amount (g)	Purity (%)	Compound	Amount (g)	Purity (%)
Tetracosanoic	0.0153	99.9	Myristic	0.0183	99.9
Docosanoic	0.0201	99.8	Dodecanoic	0.0174	99.7
Eicosanoic	0.0232	99.4	Decanoic	0.0200	99.9
Octadecanoic	0.0249	99.6	Octanoic	0.0389	99.9
Palmitic	0.0159	99.8	Hexanoic	0.0253	99.9

7-29-14 [redacted]

(Continue on 61478)

Name	Project No.	Location	61478
[redacted]	RP1581		Date: 5/19/14

Standard Prep (continued from 61468)

- To the tetracosanoic acid material, 6.00 ml of IPA (Fisher, Lot # 132647) was added the solution was vortexed mixed. Not all of the solid went into solution. Looking up the solubility data for this material, THF and chloroform were mentioned as good solvents. About 2.55 ml of THF (Honeywell, Lot # DA 191) was added to the IPA solution, and the remaining solid material went into solution. The resulting solvent composition was roughly 30% THF in IPA.

$$[\text{Tetracosanoic Acid}] = \frac{0.0153\,g \times 0.999}{8.55\,mL_{THF:IPA}} \times \frac{1\times10^6\,\mu g}{g} = \underline{1788\ \tfrac{\mu g}{mL}}$$

-

- For the docosanoic acid material 2.00 ml of THF was added, followed by 4.666 ml of IPA, and the solution was vortexed mixed. All of this solid material went into solution.

$$[\text{Docosanoic Acid}] = \frac{0.0201\,g \times 0.998}{6.666\,mL_{THF:IPA}} \times \frac{1\times10^6\,\mu g}{g} = \underline{3009\ \tfrac{\mu g}{mL}}$$

A 30% THF in IPA soln will be prepared and the remaining neat materials will be dissolved in 6.00 ml of this soln.

5/20/14

Diluent: Combined 30 ml of THF with 70 ml of IPA.

For the eicosanoic, octadecanoic and palmitic material, 6 ml of the diluent was added to each vial, which was then vortex mixed and sonicated for 5 min. All of the solid material for each compound went into solution.

$$[\text{Eicosanoic Acid}] = \frac{0.0232\,g \times 0.994}{6.00\,mL\ \text{diluent}} \times \frac{1\times10^6\,\mu g}{g} = \underline{3843\ \tfrac{\mu g}{mL}}$$

$$[\text{Octadecanoic Acid}] = \frac{0.0249\,g \times 0.996}{6.00\,mL\ \text{diluent}} \times \frac{1\times10^6\,\mu g}{g} = 4133\ \tfrac{\mu g}{mL}$$

7-29-14 [redacted]

Name	Project No. Location	61479
[redacted]	RP1581	Date: 5/20/14

Standard Prep

$$[\text{Palmitic Acid}] = \frac{0.0159\,g \times 0.998}{6.00\,ml} \times \frac{1\times10^6\,\mu g}{g} = 2645\ \frac{\mu g}{ml}$$

For the remaining 5 compounds, 6 ml of the diluent was added to each vial and the resulting solns were vortex mixed and sonicated for 5 min. All of the solid material, except for ~~in the~~ octanoic and hexanoic acid, went into solution. DH 5/20/14 Octanoic and hexanoic were liquids at room temp and they too went into solution.

$$[\text{Myristic Acid}] = \frac{0.0183\,g \times 0.999}{6.00\,ml\ \text{diluent}} \times \frac{1\times10^6\,\mu g}{g} = 3047\ \frac{\mu g}{ml}$$

$$[\text{Dodecanoic Acid}] = \frac{0.0174\,g \times 0.99\cancel{9}7}{6.00\,ml\ \text{diluent}} \times \frac{1\times10^6\,\mu g}{g} = 28\cancel{97}1\ \frac{\mu g}{ml}$$

7 DH 5/27/14 — DH 5/27/14

$$[\text{Decanoic Acid}] = \frac{0.0200\,g \times 0.999}{6.00\,ml\ \text{diluent}} \times \frac{1\times10^6\,\mu g}{g} = 3330\ \frac{\mu g}{ml}$$

$$[\text{Octanoic Acid}] = \frac{0.0389\,g \times 0.999}{6.00\,ml\ \text{diluent}} \times \frac{1\times10^6\,\mu g}{g} = 6477\ \frac{\mu g}{ml}$$

$$[\text{Hexanoic Acid}] = \frac{0.0253\,g \times 0.999}{6.00\,ml\ \text{diluent}} \times \frac{1\times10^6\,\mu g}{g} = 4212\ \frac{\mu g}{ml}$$

5/27/14

Balance: AY338

✓ wt set: EPA-K-5
20 mg => 0.0200 g
1 g => 1.0000 g
20 g => 20.0005 g

Fatty Acids Kit #2, Supelco, Lot # LC06978

The following acid analytes were placed in tared, amber vials:

Acid Analyte	Purity %	Amount (g)	Analyte	Purity (%)	Amount (g)
Palmitoleic	99.9	0.0307	Eicosatetraenoic (cis-5,8,11,14)	99.9	0.0384
Docosahexaenoic (cis-4,7,10,13,16,19)	99.9	0.033~~2~~4 DH 5/27/14	Elaidic	99.9	0.0313
Oleic	99.7	0.0313	Nervonic	99.9	0.0360
Linoleic	99.9	0.03~~59~~61 DH 5/27/14	Erucic	99.6	0.0328
Linolenic	99.9	0.0329	Petroselinic	99.9	0.0342

[redacted] 7-29-14

Name	Project No.	Location	61481
[redacted]	RP1581		Date: 5/27/14

To each vial, 6 ml of a 30% THF in IPA soln was added, and the resulting solution was vortex mixed, and sonicated for 5 min.

[Palmitoleic Acid] = $\frac{0.0307g \times 0.999}{6.00 ml_{diluent}} \times \frac{1\times10^6 \mu g}{g} = \underline{\underline{5112}} \frac{\mu g}{ml}$

[Docosahexaenoic Acid] (cis-4,7,10,13,16,19) = $\frac{0.0334g \times 0.999}{6.00 ml_{diluent}} \times \frac{1\times10^6 \mu g}{g} = \underline{\underline{5561}} \frac{\mu g}{ml}$

[Oleic Acid] = $\frac{0.0313g \times 0.997}{6.00 ml_{diluent}} \times \frac{1\times10^6 \mu g}{g} = \underline{\underline{5201}} \frac{\mu g}{ml}$

[Linoleic Acid] = $\frac{0.0361g \times 0.999}{6.00 ml_{diluent}} \times \frac{1\times10^6 \mu g}{g} = \underline{\underline{6011}} \frac{\mu g}{ml}$

[Linolenic Acid] = $\frac{0.0329g \times 0.999}{6.00 ml_{diluent}} \times \frac{1\times10^6 \mu g}{g} = \underline{\underline{5478}} \frac{\mu g}{ml}$

[Eicosatetraenoic Acid] (cis-5,8,11,14) = $\frac{0.0384g \times 0.999}{6.00 ml_{diluent}} \times \frac{1\times10^6 \mu g}{g} = \underline{\underline{6394}} \frac{\mu g}{ml}$

[Elaidic Acid] = $\frac{0.0313g \times 0.999}{6.00 ml_{diluent}} \times \frac{1\times10^6 \mu g}{g} = \underline{\underline{5211}} \frac{\mu g}{ml}$

[Nervonic Acid] = $\frac{0.0360g \times 0.999}{6.00 ml~~\text{content}~~ \text{diluent}} \times \frac{1\times10^6 \mu g}{g} = \underline{\underline{5994}} \frac{\mu g}{ml}$ DB 5/27/14

[Erucic Acid] = $\frac{0.0328g \times 0.99~~8~~\,9}{6.00 ml_{diluent}} \times \frac{1\times10^6 \mu g}{g} = \underline{\underline{5445}} \frac{\mu g}{ml}$ DB 5/27/14

[Petroselinic Acid] = $\frac{0.0342g \times 0.999}{6.00 ml_{diluent}} \times \frac{1\times10^6 \mu g}{g} = \underline{\underline{5694}} \frac{\mu g}{ml}$

7-29-14
[redacted]

Name	Project No.	Location	60290
[redacted]	RP1581		Date: 5/12/14

Checking Methanol (MeOH) calibration set up on 2/13/14 (see pgs 60281 - 60283)
these pgs show cal ranges and set up, as well as standard preps

Using matrix modifying solution (MMS) prepared 12/6/11
(180g NaCl w/ 500 mL H_2O)

Using Methanol-d3 as internal standard for methanol analysis

Making standards: Using methylene Chloride as solvent B&J lot# CG132
Using 6mL volumetric pippette & 100 uL Drummond pipette
Balance # ~~4575~~ 4338 verified
AH 5/12/14

4-methylcyclohexanemethanol (MCHM)
TCI
Lot # RCGNA-PT >98.0%

$$\frac{0.0971\,g}{6\,mL} \times 0.98 \times \frac{1.0 \times 10^6\,ug}{1g} = 15,859.6 \rightarrow 15900\,ug/mL$$

Dimethyl 1,4-cyclohexanedicarboxylate (DM-1,4DC)
Aldrich
Lot # MKBJ2148V 97%

$$\frac{0.1125\,g}{6\,mL} \times 0.97 \times \frac{1.0 \times 10^6\,ug}{1g} = 18,187.5 \rightarrow 18200\,ug/mL$$

1,4-cyclohexanedimethanol (1,4-CHDM)
Aldrich
Lot # MKBP6651V 99%
Solid used spatula

$$\frac{0.0673\,g}{6\,mL} \times 0.99 \times \frac{1.0 \times 10^6\,ug}{1g} = 11104.5 \rightarrow 11,100\,ug/mL$$

Name	Project No.	Location	60291
[redacted]	RP1581		Date: 5/12/14

Prep Stds (cont.)

Propylene glycol phenyl ether (PPH)
Aldrich
Lot # MKBK0424V ≥ 93%

$$\frac{0.1040\,g}{6mL} \times 0.93 \times \frac{1.0 \times 10^6\,\mu g}{1g} = 16{,}120\ \mu g/mL$$

Dipropylene glycol phenyl ether (DiPPH)
Dow Chemical
Lot #: 200602920-14

$$\frac{0.1059\,g}{6mL} \times \frac{1.0 \times 10^6\,\mu g}{1g} = 17{,}650\ \mu g/mL$$

Cyclohexanol (Surrogate)
Aldrich
Lot#: 11107CH 99%

$$\frac{0.0949\,g}{6mL} \times 0.99 \times \frac{1.0 \times 10^6\,\mu g}{1g} = 15{,}658.5 \rightarrow 15{,}700\ \mu g/mL$$

DFTPP
Supelco
1000 µg/mL in acetone
Lot# LB91234

Name	Project No. Location	60292
[redacted]	RP1581	Date: 5/12/14

Making a mix standard @ 1000 ug/mL w/ all stds except cyclohexanol & DFTPP

MCHM

$$V = \frac{(1000\,\mu g/mL)(10\,mL)}{15900\,\mu g/m} = 0.629\,mL \rightarrow 629\,\mu L$$

DM-1,4 DC

$$V = 0.549\,mL \rightarrow 549\,\mu L$$

1,4-CHDM

$$V = 0.901\,mL \rightarrow 901\,\mu L$$

PPH

$$V = 0.620\,mL \rightarrow 620\,\mu L$$

DiPPH

$$V = 0.5665\,mL \rightarrow 567\,\mu L$$

Cyclohexanol 1000 ug/mL std

$$V = \frac{(1000\,\mu g/mL)(5\,mL)}{15{,}700\,\mu g/mL} = 0.318\,mL \rightarrow 318\,\mu L$$

Cal curve

Cal Pt	Conc. (ug/mL)	Amt Std (uL)	Amt Surrogate* (uL)	Amt MeCl2 (uL)	Total Volume (uL)
1	0.5	1	AH 5/12/14 ~~50~~ 100	AH 5/12/14 ~~1949~~ 1899	2000
2	1	2	AH 5/12/14 100	1898	2000
3	3	3	50	947	1000
4	5	5		945	
5	10	10		940	
6	50	50		900	
7	100	100		850	
8	200	200		750	

* Cyclohexanol a surrogate @ 50 ug/mL

[redacted] 5/12/14

EASTMAN

SDSUS / EN / 05
Version: 2.0
Revision date: 08/18/2011
Initiator: 0001
150000014291

SAFETY DATA SHEET

SECTION 1: Identification of the substance/mixture and of the company/undertaking

1.1 Product identifier
Product name: Crude MCHM

Product No.: EAN 972790. 18717-00, P1871700, P18717EA, P18717ET, P18717YZ

1.2 Relevant identified uses of the substance or mixture and uses advised against
Identified uses: Industrial chemical. gasoline blending
Uses advised against: None known.

1.3 Details of the supplier of the safety data sheet
Manufacturer / Supplier
Eastman Chemical Company
200 South Wilcox Drive
Kingsport, TN 37660-5280 US
+14232292000

Visit our website at www.EASTMAN.com or email emnmsds@eastman.com

1.4 Emergency telephone number:
For emergency health, safety, and environmental information, call 1-423-229-4511 or 1-423-229-2000.

For emergency transportation information, in the United States: call CHEMTREC at 800-424-9300 or call 423-229-2000.

SECTION 2: Hazards identification

WARNING!
HARMFUL IF SWALLOWED
CAUSES SKIN AND EYE IRRITATION
AT ELEVATED TEMPERATURES, VAPOR MAY CAUSE IRRITATION OF EYES AND RESPIRATORY TRACT

SECTION 3: Composition/information on ingredients

3.1 / 3.2 Substances / Mixtures

General information:

Chemical name	Concentration	Additional identification	Notes
4-methylcyclohexanemethanol	68 - 89%	CAS-No.: 34885-03-5 EC No.: 609-038-8	
4-(methoxymethyl)cyclohexanemethanol	4 - 22%	CAS-No.: 98955-27-2	

water	4 - 10%	CAS-No.: 7732-18-5 EC No.: 231-791-2	
methyl 4-methylcyclohexanecarboxylate	5%	CAS-No.: 51181-40-9	
dimethyl 1,4-cyclohexanedicarboxylate	1%	CAS-No.: 94-60-0 EC No.: 202-347-5	
methanol	1%	CAS-No.: 67-56-1 EC No.: 200-659-6 INDEX No.: 603-001-00-X	#
1,4-cyclohexanedimethanol	1 - 2%	CAS-No.: 105-08-8 REACH Registration No.: 01-2119448337-34-0000 01-2119448337-34-0002	#

* All concentrations are percent by weight unless ingredient is a gas. Gas concentrations are in percent by volume.
# This substance has workplace exposure limit(s).
PBT: persistent, bioaccumulative and toxic substance.
vPvB: very persistent and very bioaccumulative substance.

SECTION 4: First aid measures

4.1 Description of first aid measures

Inhalation: Treat symptomatically. Move to fresh air. Get medical attention if symptoms persist.

Eye contact: Immediately flush with plenty of water for at least 15 minutes. If easy to do, remove contact lenses. Get medical attention. In case of irritation from airborne exposure, move to fresh air. Get medical attention if symptoms persist.

Skin contact: Immediately flush with plenty of water for at least 15 minutes while removing contaminated clothing and shoes. Get medical attention immediately. Wash contaminated clothing before reuse. Destroy or thoroughly clean contaminated shoes.

Ingestion: Call a physician or poison control center immediately. Only induce vomiting at the instruction of medical personnel. Never give anything by mouth to an unconscious person. Not relevant, due to the form of the product.

4.2 Most important symptoms and effects, both acute and delayed: No data available.

4.3 Indication of any immediate medical attention and special treatment needed

Hazards: No data available.

Treatment: Treat symptomatically.

SECTION 5: Firefighting measures

General fire hazards: None known.

5.1 Extinguishing media

Suitable extingishing media: Water spray. Dry chemical. Carbon Dioxide. Alcohol foam.

Unsuitable extinguishing media: None known.

5.2 Special hazards arising from the substance or mixture: None known.

5.3 Advice for firefighters

Special Fire Fighting Procedures: Fight fire from a protected location.

Special protective equipment for fire-fighters: Self-contained breathing apparatus and full protective clothing must be worn in case of fire.

SECTION 6: Accidental release measures

6.1 Personal precautions, protective equipment and emergency procedures: Wear appropriate personal protective equipment.

6.2 Environmental precautions: Avoid release to the environment.

6.3 Methods and material for containment and cleaning up: Absorb spill with vermiculite or other inert material, then place in a container for chemical waste. Large Spillages: Flush spill area with water spray. Prevent runoff from entering drains, sewers, or streams. Dike for later disposal.

Notification Procedures: In the event of a spill or accidental release, notify relevant authorities in accordance with all applicable regulations.

SECTION 7: Handling and storage:

7.1 Precautions for safe handling: Avoid breathing vapor from heated material. Avoid contact with eyes, skin, and clothing. Do not taste or swallow. Use only with adequate ventilation. Wash thoroughly after handling.

7.2 Conditions for safe storage, including any incompatibilities: Keep container closed. Keep away from food, drink and animal feedingstuffs.

7.3 Specific end use(s): Industrial chemical. gasoline blending

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SECTION 8: Exposure controls/personal protection

8.1 Control parameters

Occupational exposure limits

If exposure limits have not been established, maintain airborne levels to an acceptable level.

Chemical name	Type	Exposure Limit values		Source
methanol	TWA	200 ppm		US. ACGIH Threshold Limit Values (01 2010)
	STEL	250 ppm		US. ACGIH Threshold Limit Values (01 2010)
	PEL	200 ppm	260 mg/m3	US. OSHA Table Z-1 Limits for Air Contaminants (29 CFR 1910.1000) (02 2006)

Biological limit values

Chemical name	Exposure Limit values	Source
methanol (methanol: Sampling time: End of shift.)	15 mg/l (Urine)	ACGIH BEL (01 2010)

8.2 Exposure controls

Appropriate engineering controls: Good general ventilation (typically 10 air changes per hour) should be used. Ventilation rates should be matched to conditions. If applicable, use process enclosures, local exhaust ventilation, or other engineering controls to maintain airborne levels below recommended exposure limits. If exposure limits have not been established, maintain airborne levels to an acceptable level.

Individual protection measures, such as personal protective equipment

General information: Eye bath. Safety shower. Washing facilities.

Eye/face protection: Wear safety glasses with side shields (or goggles). Wear a full-face respirator, if needed.

Skin protection

Hand protection: Wear chemical-resistant gloves, footwear, and protective clothing appropriate for the risk of exposure. Contact health and safety professional or manufacturer for specific information.

Other: No data available.

Respiratory Protection: If engineering controls do not maintain airborne concentrations below recommended exposure limits (where applicable) or to an acceptable level (in countries where exposure limits have not been established), an approved respirator must be worn. In the United States of America, if respirators are used, a program should be instituted to assure compliance with OSHA Standard 63 FR 1152, January 8, 1998. Respirator type: Air-purifying respirator with an appropriate, government approved (where applicable), air-purifying filter, cartridge or canister. Contact health and safety professional or manufacturer for specific information.

Hygiene measures: Observe good industrial hygiene practices.

Environmental Controls: No data available.

SECTION 9: Physical and chemical properties

9.1 Information on basic physical and chemical properties

Appearance	
Physical State:	Liquid
Form:	Liquid
Color:	Colorless
Odor:	Alcohol
Odor Threshold:	No data available.
pH:	No data available.
Freezing Point:	0 °C
Boiling Point:	180 °C
Flash Point:	112.8 °C (Setaflash Closed Cup)
Evaporation Rate:	No data available.
Flammability (solid, gas):	No data available.
Flammability Limit - Upper (%)–:	No data available.
Flammability Limit - Lower (%)–:	No data available.
Vapor pressure:	No data available.
Vapor density (air=1):	No data available.
Relative density:	< 1 (estimated)
Solubility(ies)	
Solubility in Water:	Appreciable
Solubility (other):	No data available.
Partition coefficient (n-octanol/water):	No data available.
Autoignition Temperature:	No data available.
Decomposition Temperature:	Thermal stability not tested. Low stability hazard expected at normal operating temperatures.
Viscosity:	No data available.
Explosive properties:	No data available.
Oxidizing properties:	No data available.

SECTION 10: Stability and reactivity

10.1 Reactivity: Materials containing similar structural groups are normally stable.

10.2 Chemical stability: Not fully evaluated.

10.3 Possibility of hazardous reactions: None known.

10.4 Conditions to avoid: Excessive heat.

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10.5 Incompatible materials:	Strong oxidizing agents.
10.6 Hazardous decomposition products:	Carbon Dioxide. Carbon Monoxide.

SECTION 11: Toxicological information

Information on likely routes of exposure

Inhalation:	At elevated temperatures, vapor may cause irritation of eyes and respiratory tract.
Ingestion:	Harmful if swallowed.
Skin contact:	Causes skin irritation.
Eye contact:	Causes serious eye irritation.

11.1 Information on toxicological effects

Acute Toxicity

Oral

Product:	Oral LD-50: (Rat): 825 mg/kg

Dermal

Product:	Dermal LD-50: (Rat): > 2,000 mg/kg

Inhalation

Product:	No data available.
Specified substance(s)	
4-methylcyclohexanemethanol	No data available.
4-(methoxymethyl)cyclohexanemethanol	No data available.
water	No data available.
methyl 4-methylcyclohexanecarboxylate	No data available.
dimethyl 1,4-cyclohexanedicarboxylate	No data available.
methanol	No data available.
1,4-cyclohexanedimethanol	LC50 (Rat, 6 h): > 3 mg/l (highest concentration tested)

Repeated dose toxicity

Product:	No data available.
Specified substance(s)	
4-methylcyclohexanemethanol	No data available.
4-	No data available.

(methoxymethyl)cyclohexanemethanol	
water	No data available.
methyl 4-methylcyclohexanecarboxylate	No data available.
dimethyl 1,4-cyclohexanedicarboxylate	No data available.
methanol	No data available.
1,4-cyclohexanedimethanol	NOEL - No Observable Effect Level (Rat, in drinking water, 90 d): 8000 mg/l

Skin corrosion/irritation:

Product: (Rabbit, 24 h): strong

Serious eye damage/eye irritation:

Product: (Rabbit): moderate

Respiratory or skin sensitization:

Product: Skin Sensitization:, (Guinea Pig) - Not a skin sensitizer.

Germ cell mutagenicity

In vitro

Product: No data available.

Specified substance(s)

4-methylcyclohexanemethanol	No data available.
4-(methoxymethyl)cyclohexanemethanol	No data available.
water	No data available.
methyl 4-methylcyclohexanecarboxylate	No data available.
dimethyl 1,4-cyclohexanedicarboxylate	No data available.
methanol	No data available.
1,4-cyclohexanedimethanol	Mutagenicity - Bacterial, : negative +/- activation Mutagenicity - Mammalian, : negative +/- activation Chromosomal abberation, : negative +/- activation

In vivo

Product: No data available.

Specified substance(s)

4-methylcyclohexanemethanol	No data available.
4-(methoxymethyl)cyclohexanemethanol	No data available.
water	No data available.

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methyl 4-methylcyclohexanecarboxylate	No data available.
dimethyl 1,4-cyclohexanedicarboxylate	No data available.
methanol	No data available.
1,4-cyclohexanedimethanol	No data available.

Carcinogenicity

Product: No data available.

Specified substance(s)

4-methylcyclohexanemethanol	No data available.
4-(methoxymethyl)cyclohexanemethanol	No data available.
water	No data available.
methyl 4-methylcyclohexanecarboxylate	No data available.
dimethyl 1,4-cyclohexanedicarboxylate	No data available.
methanol	No data available.
1,4-cyclohexanedimethanol	No data available.

Reproductive toxicity

Product: No data available.

Specified substance(s)

4-methylcyclohexanemethanol	No data available.
4-(methoxymethyl)cyclohexanemethanol	No data available.
water	No data available.
methyl 4-methylcyclohexanecarboxylate	No data available.
dimethyl 1,4-cyclohexanedicarboxylate	No data available.
methanol	No data available.
1,4-cyclohexanedimethanol	No data available.

Specific target organ toxicity - single exposure

Product: No data available.

Specified substance(s)

4-methylcyclohexanemethanol	No data available.
4-(methoxymethyl)cyclohexanemethanol	No data available.

water	No data available.
methyl 4-methylcyclohexanecarboxylate	No data available.
dimethyl 1,4-cyclohexanedicarboxylate	No data available.
methanol	No data available.
1,4-cyclohexanedimethanol	No data available.

Specific target organ toxicity - repeated exposure

Product: No data available.

Specified substance(s)

4-methylcyclohexanemethanol	No data available.
4-(methoxymethyl)cyclohexanemethanol	No data available.
water	No data available.
methyl 4-methylcyclohexanecarboxylate	No data available.
dimethyl 1,4-cyclohexanedicarboxylate	No data available.
methanol	No data available.
1,4-cyclohexanedimethanol	No data available.

Aspiration hazard

Product: No data available.

Specified substance(s)

4-methylcyclohexanemethanol	No data available.
4-(methoxymethyl)cyclohexanemethanol	No data available.
water	No data available.
methyl 4-methylcyclohexanecarboxylate	No data available.
dimethyl 1,4-cyclohexanedicarboxylate	No data available.
methanol	No data available.
1,4-cyclohexanedimethanol	No data available.

Other adverse effects: No data available.

SECTION 12: Ecological information

12.1 Toxicity

Acute toxicity

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Fish

Product:	LC-50 (Fathead Minnow, 96 h): 57.4 mg/l NOEC: (Fathead Minnow, 96 h): 25 mg/l

Aquatic invertebrates

Product:	EC-50 (daphnid, 48 h): 98.1 mg/l NOEC: (daphnid, 48 h): 40 mg/l

Chronic Toxicity

Fish

Product:	No data available.
Specified substance(s)	
4-methylcyclohexanemethanol	No data available.
4-(methoxymethyl)cyclohexanemethanol	No data available.
water	No data available.
methyl 4-methylcyclohexanecarboxylate	No data available.
dimethyl 1,4-cyclohexanedicarboxylate	No data available.
methanol	No data available.
1,4-cyclohexanedimethanol	No data available.

Aquatic invertebrates

Product:	No data available.
Specified substance(s)	
4-methylcyclohexanemethanol	No data available.
4-(methoxymethyl)cyclohexanemethanol	No data available.
water	No data available.
methyl 4-methylcyclohexanecarboxylate	No data available.
dimethyl 1,4-cyclohexanedicarboxylate	No data available.
methanol	No data available.
1,4-cyclohexanedimethanol	No data available.

Toxicity to Aquatic Plants

Product:	No data available.
Specified substance(s)	
4-methylcyclohexanemethanol	No data available.
4-(methoxymethyl)cyclohexan	No data available.

emethanol	
water	No data available.
methyl 4-methylcyclohexanecarboxylate	No data available.
dimethyl 1,4-cyclohexanedicarboxylate	No data available.
methanol	No data available.
1,4-cyclohexanedimethanol	EC-50 (Alga, 72 h): > 122.9 mg/l (only concentration tested) NOEC: (Alga, 72 h): >= 122.9 mg/l (only concentration tested)

12.2 Persistence and degradability

Biodegradation

Product:	No data available.
Specified substance(s)	
4-methylcyclohexanemethanol	No data available.
4-(methoxymethyl)cyclohexanemethanol	No data available.
water	No data available.
methyl 4-methylcyclohexanecarboxylate	No data available.
dimethyl 1,4-cyclohexanedicarboxylate	No data available.
methanol	No data available.
1,4-cyclohexanedimethanol	99.2 % (28 d, Ready Biodegradability: DOC Die Away Test) Readily biodegradable

Biological Oxygen Demand:

Product	BOD-5: 70 mg/g BOD-20: 1,300 mg/g

Chemical Oxygen Demand:

Product	2,450 mg/g

BOD/COD ratio

Product	No data available.
Specified substance(s)	
4-methylcyclohexanemethanol	No data available.
4-(methoxymethyl)cyclohexanemethanol	No data available.
water	No data available.
methyl 4-methylcyclohexanecarboxylate	No data available.
dimethyl 1,4-cyclohexanedicarboxylate	No data available.
methanol	No data available.

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1,4-cyclohexanedimethanol	No data available.

12.3 Bioaccumulative potential

Product:	No data available.
Specified substance(s)	
4-methylcyclohexanemethanol	No data available.
4-(methoxymethyl)cyclohexanemethanol	No data available.
water	No data available.
methyl 4-methylcyclohexanecarboxylate	No data available.
dimethyl 1,4-cyclohexanedicarboxylate	No data available.
methanol	No data available.
1,4-cyclohexanedimethanol	No data available.

12.4 Mobility in soil: No data available.

Known or predicted distribution to environmental compartments

4-methylcyclohexanemethanol	No data available.
4-(methoxymethyl)cyclohexanemethanol	No data available.
water	No data available.
methyl 4-methylcyclohexanecarboxylate	No data available.
dimethyl 1,4-cyclohexanedicarboxylate	No data available.
methanol	No data available.
1,4-cyclohexanedimethanol	0.499 - 1.6 (QSAR model)

12.5 Results of PBT and vPvB assessment: No data available.

4-methylcyclohexanemethanol	No data available.
4-(methoxymethyl)cyclohexanemethanol	No data available.
water	No data available.
methyl 4-methylcyclohexanecarboxylate	No data available.
dimethyl 1,4-cyclohexanedicarboxylate	No data available.
methanol	No data available.
1,4-cyclohexanedimethanol	Not fulfilling PBT (persistent/bioaccumulative/toxic) criteria

12.6 Other adverse effects: No data available.

SECTION 13: Disposal considerations

13.1 Waste treatment methods

General information: No data available.

Disposal Methods: Dispose of waste and residues in accordance with local authority requirements. Mix with compatible chemical which is less flammable and incinerate. Since emptied containers retain product residue, follow label warnings even after container is emptied. Residual vapors may explode on ignition; do not cut, drill, grind, or weld on or near this container.

SECTION 14: Transport information

Important Note: Shipping descriptions may vary based on mode of transport, quantities, package size, and/or origin and destination. Consult your company's Hazardous Materials/Dangerous Goods expert for information specific to your situation.

DOT

Class not regulated

IMDG - International Maritime Dangerous Goods Code

Class not regulated

IATA

Class not regulated

SECTION 15: Regulatory information

15.1 Safety, health and environmental regulations/legislation specific for the substance or mixture:

This product has been classified in accordance with hazard criteria of the Controlled Products Regulations and the MSDS contains all the information required by the Controlled Products Regulations.
WHMIS (Canada) Status: controlled
WHMIS (Canada) Hazard Classification: D/2/B

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SARA 311-312 Hazard Classification(s):
immediate (acute) health hazard

US EPCRA (SARA Title III) Section 313 - Toxic Chemical List
METHANOL

OSHA: hazardous

TSCA (US Toxic Substances Control Act): All components of this product are listed on the TSCA inventory. Any impurities present in this product are exempt from listing.

DSL (Canadian Domestic Substances List) and CEPA (Canadian Environmental Protection Act): One or more components of this product are not listed on the DSL. In Canada, its use is restricted to research and development purposes only.

MITI (Japanese Handbook of Existing and New Chemical Substances): One or more components or reactants of this product are not listed in the Handbook. In Japan, its use is restricted to research and development purposes only.

ECL (Korean Toxic Substances Control Act): One or more components of this product are not listed on the Korean inventory. In Korea, its use is restricted to research and development purposes only.

SECTION 16: Other information

HMIS® Hazard Ratings: Health - 2, Flammability - 1, Chemical Reactivity - 0

HMIS® rating involves data interpretations that may vary from company to company. They are intended only for rapid, general identification of the magnitude of the specific hazard. To deal adequately with the safe handling of this material, all the information contained in this MSDS must be considered.

Revision Information:	Not relevant.
Key literature references and sources for data:	No data available.
Training information:	No data available.
Issue Date:	08/18/2011
SDS No:	
Disclaimer:	This information is provided without warranty. The information is believed to be correct. This information should be used to make an independent determination of the methods to safeguard workers and the environment.

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Group	Analyte	CAS No.	MF	MW	# of C=C bonds	M.P. (°C)	B.P. (°C)
Saturated Fatty Acids	Hexanoic Acid (Caproic)	142-62-1	$C_6H_{12}O_2$	116.16	0	-3	205
	Octanoic Acid (Caprylic)	124-07-2	$C_8H_{16}O_2$	144.21	0	16	239
	Decanoic Acid (Capric)	334-48-5	$C_{10}H_{20}O_2$	172.26	0	32	270
	Dodecanoic Acid (Lauric)	143-07-7	$C_{12}H_{24}O_2$	200.32	0	46	296
	Tetradecanoic Acid (Myristic)	544-63-8	$C_{14}H_{28}O_2$	228.37	0	55	326
	Hexadecanoic Acid (Palmitic)	57-10-3	$C_{16}H_{32}O_2$	256.42	0	63	351
	Octadecanoic Acid (Stearic)	57-11-4	$C_{18}H_{36}O_2$	284.48	0	69	361
	Eicosanoic Acid (Arachidic)	506-30-9	$C_{20}H_{40}O_2$	312.53	0	77	376
	Docosanoic Acid (Behenic)	112-85-6	$C_{22}H_{44}O_2$	340.58	0	82	392
	Tetracosanoic Acid	557-59-5	$C_{24}H_{48}O_2$	368.64	0	82	272 (10 mm Hg)
Unsaturated Fatty Acids	Palmitoleic Acid	373-49-9	$C_{16}H_{30}O_2$	254.40	1	0.5	364
	Linolenic Acid	463-40-1	$C_{18}H_{30}O_2$	278.43	3	-11	444
	Linoleic Acid	60-33-3	$C_{18}H_{32}O_2$	280.45	2	-5	361
	Elaidic Acid *(trans isomer of Oleic acid)*	112-79-8	$C_{18}H_{34}O_2$	282.46	1	44	360
	Oleic Acid	112-80-1	$C_{18}H_{34}O_2$	282.46	1	13	360
	Petroselinic Acid	593-39-5	$C_{18}H_{34}O_2$	282.46	1	30	399
	cis-5,8,11,14-Eicosatetraenoic Acid	506-32-1	$C_{20}H_{32}O_2$	304.47	4	-49	407
	Docosahexaenoic Acid (cis-4,7,10,13,16,19)	6217-54-5	$C_{22}H_{32}O_2$	328.49	6	-44	447
	Erucic Acid	112-86-7	$C_{22}H_{42}O_2$	338.57	1	30	358
	Nervonic Acid	506-37-6	$C_{24}H_{46}O_2$	366.62	1	43	479
Saturated Fatty Acid Methyl Ester	Octanoic acid methyl ester	111-11-5	$C_9H_{18}O_2$	158.24	0		195
	Decanoic acid methyl ester	110-42-9	$C_{11}H_{22}O_2$	186.29	0	-14	224
	Dodecanoic acid methyl ester	111-82-0	$C_{13}H_{26}O_2$	214.34	0	5.2	263
	Tridecanoic Acid methyl ester	1731-88-0	$C_{14}H_{28}O_2$	228.37	0		290
	Tetradecanoic acid methyl ester	124-10-7	$C_{15}H_{30}O_2$	242.40	0	20	323
	Pentadecanoic Acid methyl ester	7132-64-1	$C_{16}H_{32}O_2$	256.42	0	19	97 (0.05 torr)
	Hexadecanoic acid methyl ester	112-39-0	$C_{17}H_{34}O_2$	270.45	0	28	332
	Heptadecanoic Acid methyl ester	1731-92-6	$C_{18}H_{36}O_2$	284.48	0		337
	Octadecanoic acid methyl ester	112-61-8	$C_{19}H_{38}O_2$	298.50	0	39	355
	Eicosanoic Acid methyl ester	1120-28-1	$C_{21}H_{42}O_2$	326.56	0		375
	Docosanoic Acid methyl ester	929-77-1	$C_{23}H_{46}O_2$	354.61	0		398
Unsaturated Fatty Acid Methyl Ester	Myristoleic Acid methyl ester (C14)	56219-06-8	$C_{15}H_{28}O_2$	240.38	1		307
	Palmitoleic Acid methyl ester (C16)	1120-25-8	$C_{17}H_{32}O_2$	268.44	1		394
	Linoleic Acid methyl ester (C18)	112-63-0	$C_{19}H_{34}O_2$	294.47	2		373
	Linolenic Acid methyl ester (C18)	301-00-8	$C_{19}H_{32}O_2$	292.46	3		365
	Oleic Acid methyl ester (C18)	112-62-9	$C_{19}H_{36}O_2$	296.49	1	-20	352
	Elaidic Acid methyl ester (C18)	1937-62-8	$C_{19}H_{36}O_2$	296.49	1	10	220 (24 mmHg)
	cis-11-Eicosenoic Acid methyl ester (C20)	2390-09-2	$C_{21}H_{40}O_2$	324.54	1		394
	Erucic Acid methyl ester (C22)	1120-34-9	$C_{23}H_{44}O_2$	352.59	1		423